METALLURGIA

The British Journal of Metals

(INCORPORATING THE METALLURGICAL ENGINEER)

Readers are invited to submit articles for publication in the editorial pages; photographs and/or drawings suitable for reproduction are especially welcome. Contributions are paid for at the usual rates. We accept no responsibility in connection with submitted manuscript. All editorial communications should be addressed to The Editor, "Metallurgia," 31, King Street West, Manchester, 3.

PRINCIPAL CONTENTS IN THIS ISSUE:

	Page
Science and Progress	165
Simplification in British Industry	166
The Display at Farnborough	166
Rapid Oxidation of Metals and Alloys in the Presence of MoO_3. By G. W. Rathenau and J. L. Meijering. *An account of experiments in which copper and heat-resisting copper alloys, iron and heat-resisting iron alloys, nickel and nickel-chromium alloys, and silver and silver-aluminium are oxidised at elevated temperatures in the presence of MoO_3 and air.*	167–172
Corrosion Problems Arising from the Use of Magnesium Alloys in Service Aircraft. By L. F. LeBrocq., B.Sc., F.R.I.C., and H.G. Cole, B.Sc., A R.I.C. *Four examples of serious corrosion in magnesium alloys are described and methods of avoiding or inhibiting the corrosion given.*	173–175
Recent Developments of Refractories in The British Iron and Steel Industries. By W. A. Archibald, B.Sc., Ph.D.	175–176
Problems in Powder Metallurgy. By H. W. Greenwood...	176–178
Co-operative Research Activities. *The gulf that formerly existed between scientific knowledge and its application is being narrowed, particularly in industry where the application of research is doing much to improve and quicken production. Contributing to this progress are many co-operative research organisations, and the following reports will keep readers informed of the activities in some fields.*	179–196
The British Iron and Steel Research Association.	179–182
The British Cast Iron Research Association.	183–184
The British Welding Research Association.	184–185
The National Physical Laboratory.	186–187
The British Ceramic Research Association	187–188
The British Non-Ferrous Metals Research Association (Creep and Fatigue Testing Equipment in the Laboratories. By J. McKeown, D.Sc., M.I. Mech. E., F.I.M.).	189–194
The Iron and Steel Institute *At the invitation of the West of Scotland Iron and Steel Institute the Iron and Steel Institute held a Special Meeting in Glasgow and summary of the proceeding and functions is given.*	197–202
Recuperative Furnaces *The use of silicon carbide tubes lends new interest to the recuperator system.*	202–203
Current Literature	204–206
High Temperature Hardness Testing.	207—208
Staff Changes and Appointments	208
Recent Developments in Materials, Tools and Equipment	209–210

LABORATORY METHODS SUPPLEMENT.

Electronic Controller for Use with High Temperature Creep Testing Equipment. By F. Gartside, A. Eng.	211–216
Some New Indicators in various Titrimetric Processes. By A. J. Nutten, B.Sc., A.R.I.C.	216–219
Colorimetric Determination of Phosphorus in Phosphor Copper. By George Norwitz and Irwin Norwitz	219—220
Correspondence—Reproducibility of Analytical Results.	220

The articles in this Journal are indexed in *The Industrial Arts Index*

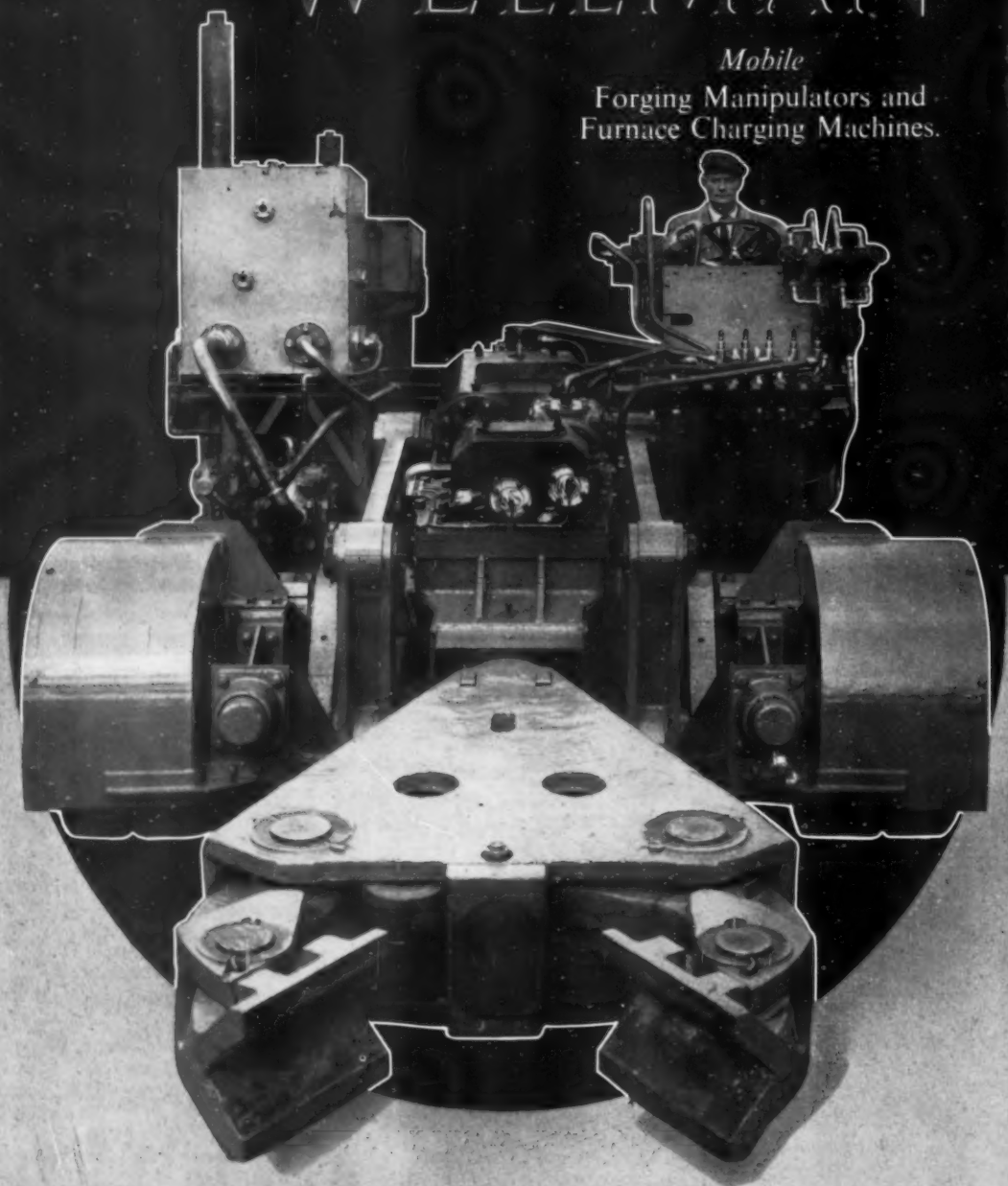
Subscription Rates throughout the World - - 24/- per annum, Post free

Published monthly by THE KENNEDY PRESS, LIMITED.

Registered Office: 31, King St. West, Manchester, 3. *Telephone:* BLA 2084. *Telegrams:* "Konpred," Manchester.
LONDON OFFICE: 21, Bedford Street, W.C.2. *Telephone:* Temple Bar 2629.

WELLMAN

Mobile
Forging Manipulators and
Furnace Charging Machines.



This Wellman Mobile Forging Manipulator is designed to handle 3 ton ingots from floor level into the reheating furnace and subsequently to withdraw them and maintain them in the desired working position under the forging hammer or press. All motions are hydraulically operated and the machine is equipped with diesel engine prime mover. This ensures complete mobility in operation. Wellman Mobile Machines are designed and constructed to operate with ease and rapidity in restricted working spaces.

MOBILE BOX CHARGERS - CHEESE & BLOOM HANDLERS - FINGER TYPE CHARGERS
PLATE HANDLERS - COIL STRIP HANDLERS - ETC.

THE WELLMAN SMITH OWEN ENGINEERING CORPORATION LIMITED
PARNELL HOUSE, WILTON ROAD, LONDON, S.W.1 WORKS: DARLASTON, SOUTH STAFFS.

METALLURGIA

THE BRITISH JOURNAL OF METALS

INCORPORATING THE "METALLURGICAL ENGINEER."

SEPTEMBER, 1950 Vol. XLII No. 251

Science and Progress

IT has been stated that human thought moves to and fro and, in some respects, repeats that of ages past and leaves the impression that what is called progress should rather be termed change. While there is much truth in this statement there can be little doubt that there is a steady and irresistible advance in that which sets the framework of our individual and social existence — the intimate knowledge of the world in which we live and its capabilities, all of which is summed up in the word science. Science knows no ebb and flow; knowledge once gained remains a permanent possession. One age may be more successful than another in adding to the store or in making full use of that which exists, but the totality of the knowledge of the world is ever on the increase. Thus understood science is ever changing the world in which we live by making us more fully acquainted with the properties and potentialities of that which we find there; it has made us see that everything in the world has special possibilities of which we can avail ourselves when the need occurs.

No sane man imagines he can foretell the future with accuracy. Who, for instance, would have given serious thought to the possibilities of the prophecies of the French writer Jules Verne when he wrote his fantastic story of the journey to the moon To-day we realise that science has made it possible to launch rockets that reach heights of 185 miles or more in a little over one minute. The speed so far achieved is in the region of two miles per second—too small as yet to overcome the gravitative pull of the earth. Apart from scientists, little thought has been given to the value of experiments in this field; so far these have confirmed the theoretical predictions that the temperature of the atmosphere at a height of 30 miles rises regularly to +50° C. and then drops to −70° C. to rise again, at a height of 75 miles, to +80° C. Interesting and positive results have also been achieved from the exploration of the sun.

Whether the results of these experiments will have an influence on practical metallurgy appears to be very doubtful, but scientific developments are gradually changing our practised ways in achieving desired results, although adherence to accustomed practices is strong and frequently can only be severed through economic necessity. Not only is the population of the world ever increasing but education is emphasising the need for higher standards of living for millions of people and, unless destruction on a large scale is coming upon us, we must progress. Science has already given indications that may lead to changes in dealing with minerals. We know, for example, that gas can be extracted from coal while in the seam and the danger of flooding mines can be stopped by freezing. Will atomic heat provide us with the means of extracting metal from the earth's crust and so limit substantially the amount and size of plant and equipment now employed?

Economic pressure will undoubtedly be the driving force in applying new developments arising from the proper assimilation of scientific discoveries and, with constantly rising costs of labour and machines, new developments, bringing about major changes, may soon be forced upon us. In this machine age, when increased or economical production is immediately associated with mechanisation, we neglect to take into account achievements, for instance, in metallurgical and electrochemistry. The extraction of base metals from the earth by chemical or electro-chemical processes might well effect a revolutionary change and cause new problems to arise which would possibly lead to a completely new basis of metal production. Modern extraction of precious metals indicates the possibilities.

The refining of platinum by electro-chemical methods provides a colloidal product which has advantages, for example, alloying and shaping by the sintering process. But colloidal metal, or mixed with other metals in a similar form to produce a desired alloy, may be used in a dry state to plate surfaces, instead of using the customary electrolytic vats. Plating by colloidal metal in the dry state is claimed to produce a more compact deposit that outlasts plating done by ordinary methods, in addition to being more economical and less difficult to apply evenly to inside hollow surfaces.

Advances in science are affecting mankind in every direction and although we are concerned here with the exploitation of scientific knowledge with a view to maintaining a progressive position for British industry, it will be appreciated that knowledge is increasing more rapidly than it can be adequately applied, because a scientific development becomes the basis of much research work before its usefulness is sufficiently known. Advances in engineering science are bound up with the development of available materials of which ferrous and non-ferrous materials play a vital part, and using, to the utmost, the resources and development in these fields will contribute materially in applying science to industry and so effect the progress desired. Much scientific and industrial research is carried out in this country on a national basis under the auspices of the Department of Scientific and Industrial Research. This Department administers funds which are used for the maintenance of certain national research establishments, and, in making grants to industrial research associations which function on a co-operation basis, by serving its members in particular fields of activity. The staffs of these Associations, in addition to carrying out research work, maintain very close contact with the industries they serve and thus assist in applying scientific knowledge with the least possible delay. Reports from some of these organisations are published elsewhere in this issue which indicate work done, in progress or contemplated. These provide practical evidence of the application of science to the solution of the ever pressing problems of industry, which, in good measure, contributes to progress.

Simplifications in British Industry

ALTHOUGH industrial conditions in Britain are very different from those which operate in the United States it is probable, in some cases, that similar methods and techniques could be introduced here with a view to raising productivity in some industries nearer to the United States level. In practically all the reports from teams visiting America to study productivity have emphasised the need for simplification in the particular industry covered. Indeed, this aspect was specially investigated by a team and a report was issued last November.[1] Since the publication of that report the group of industrialists has made an inquiry into the progress of simplification in Britain and the results are given in the present Report.[2]

The present Report contains a great deal of information of what has already been done and of what is planned in this country. It does not claim to be a comprehensive description of all that is taking place or projected, but it provides evidence that simplification is much in the minds of British industrialists. The first part deals with some of the problems raised in discussions with trade associations and individual firms in industrial centres throughout the country, following publication of the first report.

One of the most important facts brought to light was that methods of costing, when individual firms have a wide range of products, are, for the most part, inaccurate. Generally speaking the true costs of small-run and special orders are not known. Except in the comparatively few factories where such orders are segregated overhead costs are not allocated among products in their correct proportions. More accurate costing would result in the elimination of much needless and unprofitable variety.

Another important problem is that of reconciling demands from sales staff for new lines and increased variety with cost and production problems. The need is emphasised for careful market research before the adoption of any new product and also for an investigation into the suitability of the product for manufacture with existing skills and facilities. Wherever possible the adoption of a new line should be followed by the dropping of an earlier one, so that the total number is kept constant. In order to carry out this policy it is necessary for sales and production policies to be integrated at a high management level.

A misconception frequentiy arose about the effects of simplification on the consumer. There is no reason why simplification should result in lower quality standards or inadequate choice. Greater concentration by a producer on a narrower range of types does not by itself imply less total variety. Each producer is still free to make his own selection of products and the existence of consumer demand for any particular article will ensure its supply.

Other points discussed are the necessity for organisation of surveys of the possibilities of simplification—and this, it is suggested, might be undertaken by the appropriate trade associations—the part played by the designer in simplification, and the needs of export markets

The second part of the Report provides a highly condensed account of practical experience of simplification in a wide range of industries. Striking examples of reduction in types of product and the effect on costs and prices are quoted. The additional knowledge gained as a result of these discussions with British industrialists has strengthened the conviction that simplification is one of the main means available for raising productivity and lowering costs; it is not surprising, therefore, that the recommendations made in the first Report are repeated.

[1] "Simplification in Industry," *Metallurgia*, November, 1949, p. 31.
[2] "Simplification in British Industry," published by the Anglo-American Council on Productivity, 21, Tothill Street, London, S.W.1, price 1s.

Farnborough Air Show

THOSE who were fortunate enough to see the recent display of British aircraft and to examine the many examples of British engineering skill, staged at Farnborough and organised by the Society of British Aircraft Constructors, would find it difficult to restrain their enthusiasm. Despite Britain's economic burdens, there was ample evidence that her scientists and engineers still hold a lead in this field. There can be no doubt that this year's show was the envy of the many overseas visitors, who were thrilled by the exhibitions given, which greatly increased the prestige of British aircraft.

Last year, it will be remembered the Brabazon I stole the show for piston-engined types on the second day, when it made its first flight. The jet airliner, the Comet made its initial breath-taking appearance, while another of similar type, the Tudor VIII was also to be seen. Several airliners, such as the Handley-Page Hermes, the Vickers Vicount, and the Armstrong Whitworth Opollo, with their turbine driven propellers, were also on view. This year these same aircraft were to be seen; the Comet carrying the blue and white colours of B.O.A.C., while the Vicount carried the emblem of B.E.A. The latter had been on scheduled service for 25 days during which she carried nearly 2000 passengers. The Comet is due for service on Britain's Empire air route near the beginning of next year; with her jets whistling quietly, this plane gave several demonstrations at her cruising speed of 490 miles per hour.

While last year emphasis was on civil aircraft, the swing to military type this year was evident especially jet fighters. Probably the most astounding performance was that by the pilot in a Meteor who performed acrobatics over the Farnborough enclosure with a 1000-lb. bomb slung under each wing. Other jet fighters, such as the Hawker P. 1081 and the Supermarine 535 also performed amazingly. Powered by a Rolls-Royce Nene turbo-jet, with reheat equipment for additional speed in short bursts, the Hawker P.1081 thrilled the spectators, while the pilot of the Supermarine gave a 760 miles per hour run down the aerodrome. Remarkable performances were also put up by the newly released Sapphire and by the Avon. Admiration was everywhere expressed for the jet bomber, the Canberra, which the pilot handled as though it were a fighter.

Apart from the flying displays, which were magnificent, the display of materials, jet engines, rocket motors, radio sets, aircraft parts and equipment reached a very high standard and after the thrills of watching the flying it was less exhausting to examine many of these exhibits in the main exhibition hall and to note with appreciation the high degree of skill achieved.

This exhibition held at Farnborough is essentially a trade show which attracts representatives, not only from foreign airlines throughout the world but observers from military forces in overseas countries and the high total figures of the aviation products exported from Britain is a tribute to the skill and ingenuity of those engaged in the aircraft industry and to the enterprise of the firms concerned.

Rapid Oxidation of Metals and Alloys in the Presence of MoO_3

By G. W. Rathenau and J. L. Meijering

Philips Research Laboratories—N. V. Philips' Gloeil-lampenfabrieken, Eindhoven, Netherlands.

An account is given of experiments in which copper and heat-resisting copper alloys, iron and heat-resisting iron alloys, nickel and nickel-chromium alloys, and silver and silver-aluminium are oxidised at elevated temperatures in the presence of MoO_3 and air. MoO_3 promotes oxidation by the formation of liquid oxide phases in which the atoms of the basic metal can take part. Owing to the incorporation of molybdenum into the scale, the oxidation resistance may be affected below the lowest eutectic. In the case of silver alloys, MoO_3 plays an additional role, since oxidation of silver above 180° C. is possible only by complex oxide formation. The results are discussed in detail.

IN the course of measurements of the oxidation of heat-resisting alloys at 750° C. in air, it was found that the presence of a piece of molybdenum wire in the furnace had a disastrous effect. For example, an 8% aluminium bronze lost 6 mg./cm.² after 3 hours and the loss continued roughly linearly with time after the specimen had been transferred to a clean furnace. In all, a loss of 115 mg./cm.² occurred in 49 hours. Without contamination, the oxidation of this aluminium bronze is negligible at 750° C.

The effect is clearly due to the formation of MoO_3 vapour. Copper and iron themselves also proved very vulnerable to this type of attack and Fig. 1 shows the oxide scale formed on Armics iron strip heated at 735° C. in contact with MoO_3 powder *in vacuo* The oxide shows clear signs of previous melting and this is not surprising in view of the fact that the melting point of MoO_3 (795° C.) will be lowered by the iron oxides. More striking is the result of a similar experiment with OFHC-copper at 500° C.; substantial fusion of the scale had taken place.

In the meantime, a paper by Leslie and Fontana[1] appeared, describing similar "catastrophic" oxidations, especially of chromium-nickel steels *containing* molybdenum, above about 800° C. To our surprise, the possibility of liquid formation playing a part was not even mentioned, neither by the authors nor in the discussion. The explanation offered by the authors was based on the catalytic action of nascent oxygen formed by the dissociation of MoO_3 vapour.

Experimental Procedure

To confirm the idea that the formation of liquid is the important point, a systematic investigation of the oxidation of copper and 8% aluminium-bronze at temperatures between 410° C. and 550° C. was undertaken. At these temperatures both the vapour pressure of MoO_3 (about 2 mm. at 750° C.) and its dissociation are very small indeed.

It proved advantageous, as will be shown later, to carry out most of the experiments on wire specimens. The 0·5 mm. wire was passed through a furnace at 600° C. in a dried nitrogen/hydrogen mixture. After this anneal, for each penetration-depth determination, five wires of 10 cm. length were buried in MoO_3 powder in a nickel boat and heated in air at the required temperature. At the same time, for comparison, another five wires were heated in air alone, in a separate tube in the same furnace.

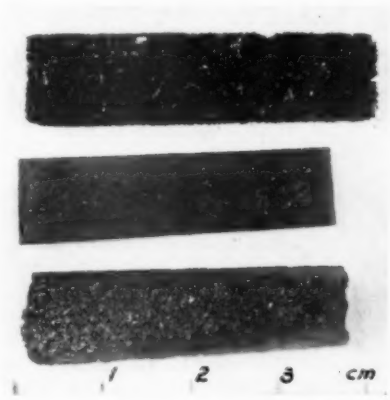


Fig. 1. — Strip of Armco iron heated for 3 hours at 735° C° in MoO_3 powder *in vacuo*. At the top: outside of the oxide layer. At the bottom: inner side of it.

The experiments with MoO_3 powder in contact with the specimens, necessitated by the negligible vapour pressure (about 10^{-5} mm. at 500° C.), would seem to be of no direct practical importance. It is thought, however that information obtained with MoO_3 is of more general interest, bearing in mind the analogous V_2O_5 and PbO. The effects of V_2O_5-containing Diesel-ashes and of PbO from leaded petrol are surely of practical importance in engineering.

After removal of the ends for metallographic and X-ray examination, some of the wires were tested in a Schopper tensile machine. Figs. 2 and 3 show the effective depth of penetration caused by the action of MoO_3:—

$$\tau_0 \left(1-\sqrt{K_M/K_A}\right)$$

where τ_0 is the original radius of the wire,
K_M is the average breaking load of the wires heated in contact with MoO_3,
and K_A is the average breaking load of the wires heated in air.

For copper, where the oxidation in air is not negligible, this formula is questionable, but for the course of the curves, this is of no consequence.

Copper and Copper Alloys

Cooling curves were taken of several mixtures of molybdenum and copper oxides (in technical nitrogen)

[1] W. C. Leslie and M. G. Fontana, *Trans. Amer. Soc. Metals*, **41**, 1213 (1949).

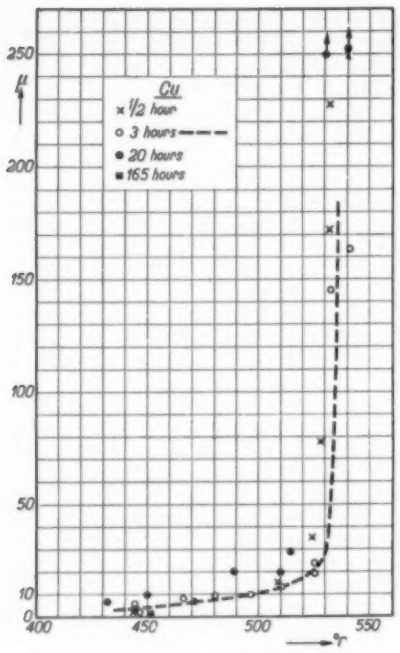


Fig. 2.—Penetration depth on oxidising 0·5 mm. copper wires in the presence of MoO_3 at different temperatures.

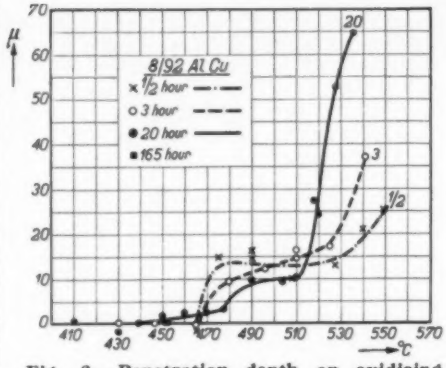


Fig. 3.—Penetration depth on oxidising 8% aluminium bronze wires in the presence of MoO_3 at different temperatures.

to determine the eutectic arrests. With CuO a eutectic at 735° C. was observed, but with Cu_2O arrests around 500° C. were noted, as is the case with mixtures of MoO_3 and other oxides of univalent metals, e.g., MoO_3 with Ag_2MoO_4 (see section on Silver and Silver Alloys), Na_2MoO_4 or K_2MoO_4.

In a silica crucible, the binary MoO_3–Cu_2O eutectic temperature was found to be 535° C.; in ternary mixtures with MoO_2, 470° C. was observed. About the same temperature, 480° C., was found with mixtures without MoO_2, but in a copper crucible which was appreciably attached. The third component in this case may, of course, be said to be MoO_2 or copper, at choice; as will be shown later, pure metallic copper can be precipitated from such oxide mixtures. Addition of Al_2O_3 powder resulted in a mere 5° C. decrease in the eutectic temperature.

As will be seen from Fig. 3, the temperature at which the influence of MoO_3 on aluminium-bronze becomes appreciable agrees very well with the lowest eutectic (i.e., 465° C.). With copper, a "tail" persists below 470° C., corresponding roughly with a doubling of the oxidation of copper in air alone.

At 535° C. liquid can be formed from MoO_3 and Cu_2O alone, so that the fluid part of the reaction zone can extend itself further outward, causing a marked acceleration in the oxidation curve of copper and, less sharply, of aluminium-bronze. The ternary eutectic does not show in Fig. 2 as it does in Fig. 3, presumably because aluminium promotes the formation of MoO_2.

A probable influence of geometrical factors was found, the attack on copper wire of 1·5 mm. diameter (instead of 0·5) was more rapid between 500° C. and 530° C.

The same general picture was shown by the curves of elongation versus temperature. Weighing experiments carried out with strip, for copper, aluminium-bronze, and ternary copper-beryllium alloys, gave 470° C. as the temperature of the onset of additional attack. For higher temperatures, the results are not quantitatively reproducible, the chief difficulties being non-uniformity of the attack as a result of drop formation and the precarious removal of the material sticking to the specimen. The tensile test measures the greatest attack, not some vague average, and cleaning of the test pieces after oxidation is unnecessary. The agreement between the five wires treated identically was, on the whole, quite good, but naturally became poorer as the severity of attack increased; for instance, of the five aluminium bronze wires heated for 3 hours at 541° C. with MoO_3, one* was attacked more than twice as much as the other four. Fig. 3 gives the results for three different batches of aluminium-bronze wire and it will be noted that good agreement was obtained. Later experiments carried out under slightly different conditions gave penetration depths somewhat lower than those of Fig. 3 but the trend of the curves remained the same. In contrast to copper, the absolute rate of attack of aluminium-bronze by MoO_3 is rather sensitive to the pre-treatment of its surface, no doubt due to the Al_2O_3 skin.

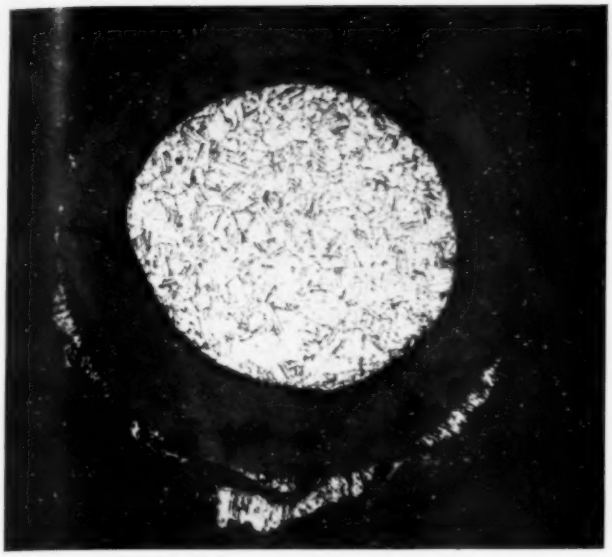
Metallographic examination supported the conclusions drawn from the tensile and weighing tests. Copper showed little preference for intercrystalline attack by liquid oxides, and aluminium-bronze still less. The surface of aluminium-bronze was rather rough after half-an-hour's attack, maybe as a result of the thin Al_2O_3 skin being broken through locally in the initial stages†. After longer times, the surface becomes smoother, so that it seems reasonable to attribute the abnormal time-dependence of the penetration depth in aluminium-bronze, in the presence of MoO_3 between 470° C. and 510° C., to a notch effect.

After oxidation above 500° C. in the presence of MoO_3, aluminium-bronze usually showed a curious phenomenon. Under an external layer consisting mainly of copper oxides, a thin layer of pure copper was seen, sometimes beautifully coherent. This copper layer and the alloy not yet attacked, were separated by a thick grey oxide layer, which Dr. Braun, of this laboratory showed by X-ray analysis to contain much MoO_2, and some CuO, Cu_2O and metallic copper.

An unidentified phase was also present, but no α- or γ-Al_2O_3 was detected. The aluminium-bronze beneath this intermediate oxide layer showed no diffusion gradient. It may also be mentioned that the copper layer was also found on aluminium-bronze heated for half-an-hour at 750° in air contaminated with MoO_3 vapour. A similar copper layer was found by Hallowes

* That shown in Fig. 4.

† A thicker layer of Al_2O_3, formed by heating aluminium bronze for 6 hours at 980° C. in moist hydrogen, was found to resist a subsequent attack by MoO_3 and air (8 hours at 500° C.) successfully, except at a spot where the Al_2O_3 layer had peeled off. Aluminium strip (99·99%) at 630° C. was not attacked by MoO_3 either, but a mixture of aluminium powder and MoO_3, on heating, gave a semi-explosive reaction.



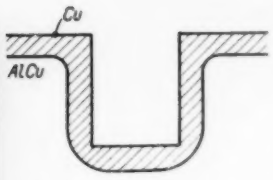


Fig. 5.
Schematic section of an aluminium bronze test-piece with sharp corners of 90° and 270° attacked by MoO_3. Copper layer follows original surface. Aluminium bronze is rounded off. Shaded area is intermediate oxide layer.

Fig. 4.
Aluminium bronze wire oxidised 3 hours at 541° C. in MoO_3, showing thick intermediate oxide layer between aluminium bronze and metallic copper. Outside : Bakelite etched with $NH_4OH + H_2O_2$ ×140

and Voce[2] in aluminium-bronze heated in steam contaminated with SO_2.

The copper layers are rough on the outside but smooth on the inside facing the aluminium-bronze. In some cases veins of copper are seen penetrating the intermediate oxide layer. The location of the smooth inner surface of the copper layer appears to coincide exactly with the original surface of the specimen. In Fig. 4, the inner diameter of the copper ring differs less than 0·01 mm. from the original wire diameter. At a 90° ridge of an aluminium-bronze specimen, a 90° corner of copper is formed, while the aluminium-bronze beneath is rounded off (see Fig. 5). Probably the Al_2O_3 skin on the original specimen determines the location of the inner copper surface.

The thin copper layers may form under circumstances where copper wires many times thicker are oxidised completely, the attack of aluminium-bronze itself, which produces the copper being much slower. It is difficult to imagine, therefore, that the copper layer is present during the reaction ; it may well be formed during the cooling to room temperature. To test this view, the reaction was observed microscopically at a magnification of 150 to 300. The aluminium-bronze wire was placed in a thin Al_2O_3 cylinder along with a thermocouple. The two were placed symmetrically with reference to an enveloping circularly arranged heating element, thus allowing an indirect approximation to the temperature to be determined. Small grains of MoO_3 placed on the flat end of the aluminium-bronze wire were seen to " melt " rapidly at about 540° C., spreading out over the surface of the aluminium-bronze and blackening it ; the visible reaction ended after a few seconds. After cooling and scratching, part of the copper layer formed under the external oxide scale became visible. Then, after nitrogen had been passed through, the apparatus was heated again. The copper coloured regions became grey, with only a slight copperish hue, but on cooling the original copper colour was restored. Why copper rather than MoO_2 should crystallise on the outside is not clear.

[2] A. P. C. Hallowes and E. Voce, *Metallurgia*, **34**, 119 (1946).

Further support for this point of view was obtained in the following manner. Three boats with aluminium-bronze wire in MoO_3 powder were heated for half-an-hour to 550° C. One was cooled directly to room temperature whilst the others were transferred to furnaces at 500° C. and 460° C., respectively, and heated for a further 40 hours. Only the last wire showed no metallic copper, presumably because this had crystallised at 460° C. (but not at 500° C.) and was oxidised subsequently.

Iron, Nickel and their Alloys

Further systematic experiments were made with 0·5 mm. wires of nickel, chromium steel (25·2% Cr 0·13% C) and 19/9 chromium-nickel steel (0·63% Nb, 0·061% C and 0·03% N). For the annealing treatments, see the captions to Figs. 6, 7 and 8.

A MoO_3-MoO_2 eutectic was observed at 778° C. Addition of Cr_2O_3 lowered it to 772° C., whilst NiO additions resulted in an arrest at 764° C. Mixtures of MoO_3 with " FeO " (a mixture of Fe_2O_3 and iron powder) formed a eutectic at 715° C. which was lowered to 705° C. by MoO_2 additions. This was the lowest eutectic found in this group of oxides. MoO_3 with 8% MoO_2 + 6% FeO + 6% Cr_2O_3, for instance, also gave 705° C. A further addition of NiO did not affect this temperature, but MoO_3 with 8% MoO_2 + 8% FeO + 8% Cr_2O_3 had an arrest at 729° C.

Experiments were also carried out with a 19/9 chromium-nickel steel crucible containing a mixture of 20g. MoO_3 with 1g. MoO_2 + 0·3g. " Feo " + 0·1g. Cr_2O_3 + 0·1g. NiO and about 3 metres of the oxidised and drawn 19/9 chromium-nickel steel test wire, cut into small pieces. This mixture was heated for 3 hours at 770° C. in air. After heating to 810° C. a cooling curve was taken showing a main arrest at 738° C. and a second one at 722° C. Corresponding experiments chromium-steel and nickel gave arrest temperatures of 728° C. and 762° C., respectively.

The determination of the effective penetration depth gave the following results. For 19/9 steel, the 3-hour and 20-hour oxidation curves (Fig. 8) bend upwards near 725° C. This temperature agrees closely with those found with the reaction mixtures. Above 770° C., near the MoO_3–MoO_2–(Cr_2O_3) eutectic temperature, a very steep rise follows, as is also the case with the 25% chromium-steel. This is to be expected since near this temperature Cr_2O_3, which should give oxidation resistance, can be fairly completely dissolved. Nickel shows a less spectacular rise near 770° C.*

In later experiments with another batch of MoO_3,

* Some experiments were carried out with nickel-chromium wire (20% Cr, 0·34% Fe) which showed a greater sensitivity to MoO_3 than nickel itself.

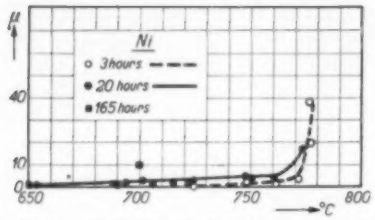


Fig. 6.—Penetration depth on oxidising nickel wires of 0·5 mm. dia. in the presence of MoO_3 at different temperatures. Pretreatment : 12 minutes 950° C. in oxygen free dried N_2/H_2 mixture.

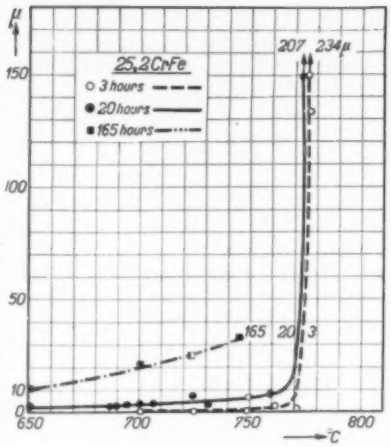


Fig. 7.—Penetration depth on 25% chromium steel wires. Pretreatment : ½-hour 980° C. in pure H_2.

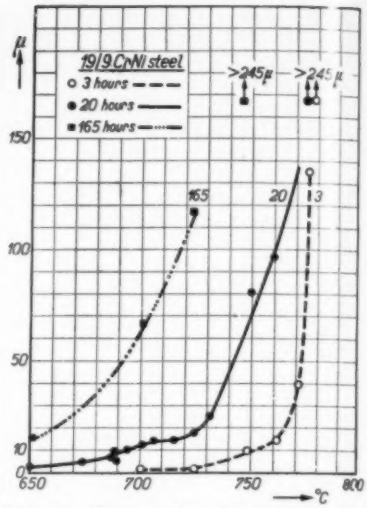


Fig. 8.—Penetration depth of 19/9 chromium-nickel steel. Pretreatment : 1 hour 1,100° C. in pure H_2.

the curves of 19/9 and chromium-steel were displaced 10° to higher temperatures.

The small " tails " of the 3-hour and 20-hour curves for nickel are not surprising, since these wires lose some of their tensile strength by intercrystalline oxidation in pure air ; this will be aggravated by the MoO_3 vapour. For instance, the 10 μ penetration after 165 hours at 700° C. is calculated from the tensile forces K_λ and K_w of 6·12 and 5·63 kg. respectively, as may be compared with their values of 8·58 and 8·57 after 3 hours at 700° C.

The persistence of an effect of MoO_3 on the oxidation of 19/9 steel and 25% chromium-steel below 705° C. is more interesting. At 650° C. and less so at 700° C., the 19/9 steel wires oxidised both with and without MoO_3, showing intercrystalline penetration. Evidently the percentage of niobium was insufficient, at least with the heat treatment applied. This is not thought to be responsible for the major part of the effect ; with 25% chromium-steel, intercrystalline oxidation does not occur.

The macroscopically homogeneous brownish oxide formed on the chromium-steel wires, after 165 hours at 650° C. with MoO_3 in air, was analysed spectroscopically. It contained about 12% FeO (or Fe_2O_3) and 6% Cr_2O_3, besides molybdenum oxide, but an unidentified X-ray structure was found containing no FeO, Fe_2O_3, Fe_3O_4, $FeO.Cr_2O_3$, or Cr_2O_3. This result suggests another means whereby MoO_3 exercises an adverse influence on oxidation resistant alloys—by spoiling an otherwise resistant oxide film by the incorporation of molybdenum oxide.

The metallographic examination of the 19/9 steel, 25% chromium-steel, nickel and nickel-chromium wires oxidised with MoO_3 revealed no metallic layers such as were found with aluminium-bronze.

Silver and Silver Alloys

Preliminary experiments had shown that silver was also severely attacked by MoO_3 in the presence of air at temperatures around 500° C. Accordingly systematic experiments were carried out on 0·5 mm. wires of silver and a silver alloy containing 4% aluminium. In the case of silver, MoO_3 plays an additional role which has not been referred to earlier. Whilst silver alone cannot oxidise in air of normal pressure above 180° C., because the dissociation pressure of Ag_2O is too high, it may do so in the presence of an oxide which stabilises Ag_2O sufficiently by the formation of a double oxide. For vanadium oxides and Cr_2O_3, this was shown by Schenck[3].

As Ag_2MoO_4 is known to melt (in air) without appreciable decomposition (at 570° C.), MoO_3 must also stabilise Ag_2O sufficiently to allow of the oxidation of silver at this and lower temperatures. Even at 750° C. in air and MoO_3 vapour, silver was oxidised rapidly*.

Cooling curves of different mixtures of Ag_2MoO_4, MoO_3 and MoO_2 (in air) gave no lower eutectic than 490° C. Al_2O_3 powder again gave a mere 5° C. depression. From Fig. 9 one would have expected 470° C. from the 20-hour curve and 510° C. from the 3-hour curve. This in itself is not too bad, nor is the persistence of marked effects below 470° C. Indeed, when no liquid is formed, MoO_3 can still influence the oxidation, because silver cannot be oxidised without this stabilising oxide. The extremely small temperature dependence at these lower temperatures (at 310° C. the effect of MoO_3 is still appreciable) remains to be explained.

Metallographic examination showed severe intercrystalline attack and disintegration into loose crystals can play an important part in addition to the oxidation of silver itself. The specimen in Fig 10. was not a wire but a special coarse-grained sample.

After 165 hours at 400° C. with MoO_3, the intercrystalline attack, though relatively less severe, is still pronounced and the persistence of the effects in Fig. 9 is partly due to this. Weighing experiments did not show this insensibility to temperature variation at low temperatures ; at 400° C. the weight loss corresponded to 1 μ for 3 hours and 2 μ for 20 hours heating time. With rising temperature, the curves of penetration depth, calculated from weighing experiments, catch up with those from tensile tests at about 510° C. (for 3 hours) and 460° C. (for 20 hours) where the latter curves are beginning to bend upwards.

Without liquid, and without appreciable vapour pressure of MoO_3, a normal intercrystalline attack is difficult to conceive. Possibly the normal reaction with the powder introduces tensile stresses which lead to intercrystalline cracks and such a mechanism may be rather insensitive to temperature. In this connection it is interesting to note that different batches of silver

3 R. Schenck, *Z. anorg. allg. Chem.*, **249**, 88 (1942).

* Silver with MoO_3 *in vacuo* was not attacked appreciably at 500° C., as was to be expected.

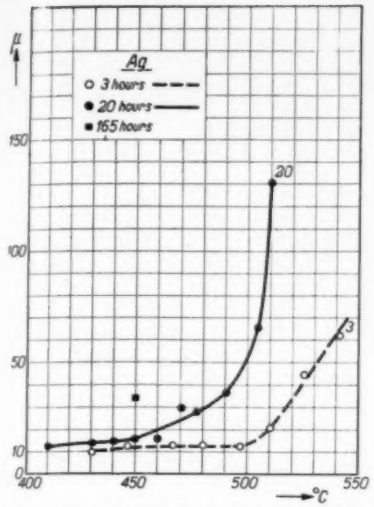


Fig. 9.—Penetration depth of 99·6% silver. Pretreatment: 12 minutes 600° C. in oxygen-free dried N_2/H_2 mixture.

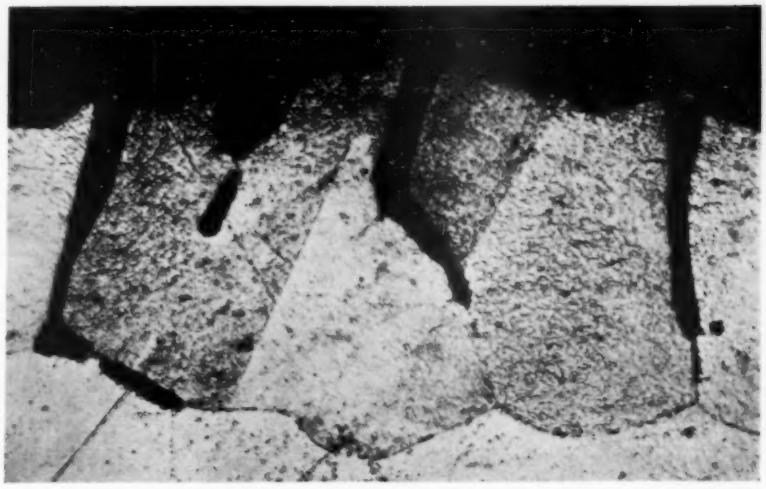


Fig. 10.—99·6% silver oxidised for 20 hours at 530° C. in MoO_3. Severe intercrystalline attack but not along twin boundaries. Etched with $NH_4OH + H_2O_2$. × 500

showed appreciably different attacks. In Fig. 9 only the results of the first batch (only 99·6% pure) are given. With four other batches, far fewer experiments were carried out but they sufficed to show a very clear correlation between attack (at 400° C.) and purity. A 99·94% pure wire gave penetration depth figures, calculated from tensile tests, of 8 μ and 14·5 μ after 20 and 165 hours at 400° C., respectively.

In the case of the alloy containing 4% aluminium (see Fig. 11) the influence of time is striking. The starting difficulties are no doubt due to the Al_2O_3 film and are probably accentuated by the fact that this film contains no silver at all, while the liquid can only be formed after Ag_2MoO_4 has been produced.

Metallographic examination showed that after half-an-hour at 540° C., hardly any of the surface of this alloy had been attacked, only a few bridgeheads, about 0·01 mm. broad being seen. Fig. 12 shows the results of 3 hours at 525° C. It suggests a eutectic structure, consisting of dark oxide lamellæ and light metallic ones. These consist of silver, not the original alloy, as was confirmed by measurement of the lattice constant. Half-an-hour at 750° C. in air saturated with MoO_3 vapour, gave a similar structure, the 0·5 mm. wire being locally transformed over the entire cross section. Below about 500° C., the eutectic nature of the picture changes to a somewhat different type (see Fig. 13).

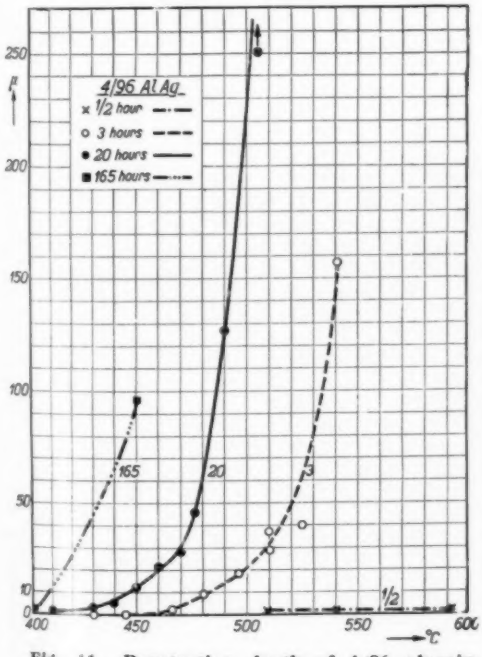


Fig. 11.—Penetration depth of 4/96 aluminium silver wires. Pretreatment: see caption to Fig. 9.

Discussion

The experimental evidence points to the conclusion

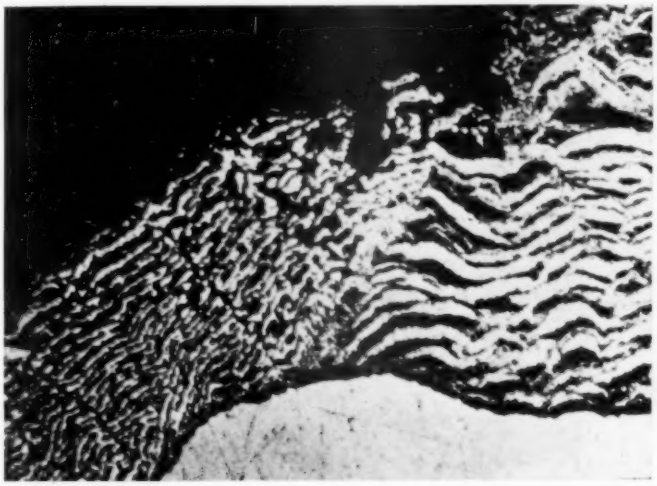


Fig. 12.—4/96 aluminium-silver oxidised for 3 hours at 525° C. in MoO_3. × 600

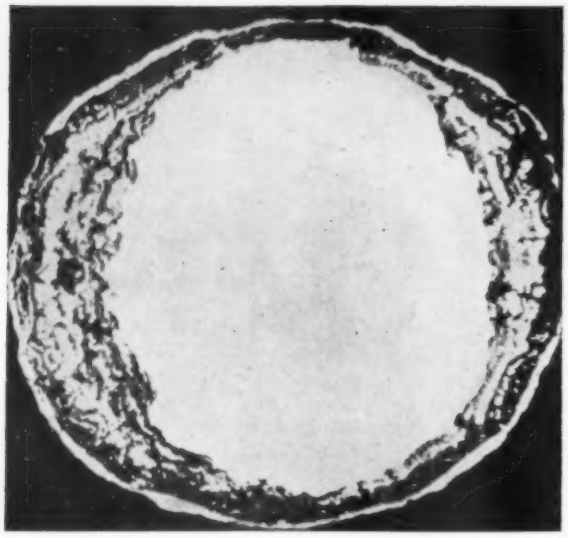


Fig. 13.—4/96 aluminium-silver oxidised for 165 hours at 451° C. in MoO_3. × 140

that the formation of a liquid oxide phase causes a rapid attack of metals and alloys in air at elevated temperatures, as has already been mentioned in an earlier note[4]. It is remarkable that in the case of aluminium-bronze it is the oxide of the base metal, copper, which is responsible for the low eutectic causing the rapid attack in the presence of molybdenum oxides and air. In the case of chromium-steel, on the other hand, the onset of heavy attack did not occur at the lowest eutectic temperature (with FeO at 705° C.), but at the temperature at which liquid molybdenum oxides dissolve the protective Cr_2O_3 layer. This difference may be explained by the amount of base metal in the oxide layers formed, and by the constitutional diagrams of the oxides involved. These factors determine whether the lowest eutectic can be formed. Cracks in the initial oxide skin of aluminium-bronze, through which the MoO_3 in this case can interact with the alloy proper, may offer another explanation. As shown in the section on copper and copper alloys, a thick layer of pure Al_2O_3, in principle, protects aluminium-bronze from rapid oxidation in the presence of MoO_3. The behaviour of 19/9 chromium nickel steel is more or less intermediate between that of chromium steel and that of aluminium-bronze.

Why does the liquid cause abnormal oxidation? Whereas it must be assumed that the oxide covering the metal is only partly liquid slightly above the temperature at which liquid first appears on heating, it seems reasonable to assume that a liquid film forms at grain boundaries of the oxide, and at the interface between metal and oxide. Diffusion and convection in a liquid are, of course, very rapid processes, allowing of speedy oxidation.

Because of the rapid diffusion and increased solubility in liquid phases, another factor mentioned earlier comes into play. Mo-ions which have already been proved to be (in the case of chromium-steel) incorporated into the scale by reaction between the solid oxides, will be taken up more quickly if part of the scale contains liquid molybdenum oxides. Molybdenum, being an element which assumes different states of ionisation at about the same energy, may easily destroy the stoichiometric character of the scale and increase oxidation in this way.

Schläpfer, Amgwerd and Preis[5, 6] studied the oxidation of heat-resisting steels, which were in contact with vanadium oxides, as well as with air. At temperatures exceeding the melting point of the vanadium oxides, these oxides greatly impaired the oxidation resistance of the alloys. Probably since, in these experiments, the added oxide leads to an oxidation of the alloys several hundred times its own weight, while the time dependence of the oxidation is about linear, the Swiss authors do not explicitly consider a liquid to be present during the whole process of oxidation. They do, however, consider liquid formation to be indispensable because of the necessary wetting of the steels by the vanadium oxides, and the formation of a porous oxide layer rich in Fe_2O_3. The continued formation of such a layer after disappearance of the liquid is attributed to the oxygen-carrying capacity of vanadium oxides. Probably these must be assumed to be concentrated near the metallic surface, or in veins running across the oxide layer. It is suggested, therefore, that liquid may continue to exist as a film between the oxide grains.

It would be interesting to carry out experiments where the reaction temperature lies initially above the eutectic temperature, but subsequently falls below it. According to the Swiss workers, one would expect the reaction to continue with abnormal velocity. Experiments, not with V_2O_5 but with MoO_3 and 19/9 chromium-nickel steel, gave the following results :—

PENETRATION DEPTH IN MICRONS

3 hr. at 779° C.	½ hr. at 779° C.	3 hr. at 750° C.	½ hr. at 779° C. + 3 hr. at 750° C.
76·5	6·2	4·3	14·9
20 hr. at 775° C.	**½ hr. at 775° C.**	**20 hr. at 750° C.**	**½ hr. at 775° C. + 20 hr. at 750° C.**
136	1·7	50	62

The experiments in the last column were carried out as follows : After the half-hour period at the higher temperature, the boats containing the wires buried in MoO_3 were transferred rapidly to an adjacent furnace at 750° C. Similar results were obtained with chromium steel.

Admittedly the results are not conclusive, but they point to the conclusion that the disappearance of the liquid results in a considerable decrease in rate of attack. Experiments with small quantities of MoO_3 are not feasible at these temperatures because of its volatility. Such experiments were tried at 550° C. with copper and aluminium-bronze, but it was not possible to obtain such large ratios between weight loss and quantity of added MoO_3 as the Swiss workers did in the case of the heat-resisting steels and V_2O_5.

The authors are greatly indebted to Messrs. W. H. Endenburg, G. de Wind, and W. Kreutzelman, for their care in carrying out the experiments described.

[4] J. L. Meijering and G. W. Rathenau, *Nature*, **165**, 240, 1950.

[5] P. Schläpfer, P. Amgwerd and H. Preis, *Schweizer Archiv*, **17**, 291 (1949).

[6] P. Amgwerd, *Eidgenössische Material prüfungs-und Versuchsanstalt*, Zürich, Bericht Nr 171, Sept., 1949.

Corrosion Problems Arising from the Use of Magnesium Alloys in Service Aircraft*

By L. F. LeBrocq, B.Sc., F.R.I.C. and H. G. Cole, B.Sc., A.R.I.C.

Metallurgy Department, Royal Aircraft Establishment, Farnborough

Four examples of serious corrosion in magnesium alloys in service aircraft are described :—(a) contact corrosion with duralumin in the presence of an electrolyte : (b) corrosion in the presence of wood under damp storage conditions : (c) corrosion of fuel tanks by wet leaded petrol, and (d) storage corrosion of welded assemblies in a 6% aluminium alloy. Methods of avoiding or inhibiting the corrosion are described.

THE low density of magnesium alloys (about 1·8) has for many years made these materials of interest to aircraft manufacturers and to others for whom lightness in a structure is of great importance. The use of magnesium alloys in aircraft has, however, been restricted by fear of corrosion troubles, a fear which is sometimes well justified, but has in other instances been much exaggerated. It is proposed to review some particular problems which have been encountered in the use of magnesium alloy parts in aircraft.

For this application the alloys are usually chromate treated and then painted, the paint normally consisting of an oil-base undercoat containing a slightly soluble pigment such as zinc chrome, to Ministry of Supply specification D.T.D. 377A, followed by a cellulose paint to any desired colour. This protective scheme usually gives reasonably good protection, but occasionally very poor results have been obtained. Some examples are given below :—

Magnesium Alloy and Duralumin in Contact

Serious corrosion occurred of an aileron which had been constructed from magnesium alloy sheet (D.T.D. 118 ; A.M. 503) riveted to duralumin stringers. Such a combination might be thought on general grounds to be unsuitable on account of the large difference in potential between the materials used (about 0·9 volts in sea-water). Nevertheless, under ordinary service conditions this combination of metals has given very little trouble.

In the particular example examined, very serious corrosion of both members of the structure had occurred wherever a de-hoar frosting fluid had been applied. Fluids of this type were used during the war to enable aircraft to take off at a moment's notice during a winter night.

On examination, the fluid was found to consist almost entirely of a strong solution of sodium nitrate in water. Laboratory examination of the fluid soon revealed that this solution had practically no effect on duralumin or magnesium alloy separately, but that very severe corrosion occurred when the metals were in contact. A notable feature of the corrosion, moreover, was the strong smell of ammonia which was produced after a short time.

To study the reaction more carefully a U-shaped glass cell was set up with duralumin in one limb and magnesium alloy (D.T.D. 118) in the other. The bottom of the U-shaped bend was plugged with a block of agar jelly containing 1% of agar and 5% of sodium nitrate, while each limb was filled with the 5% sodium nitrate solution alone. The two pieces of metal were joined by a piece of copper wire, and access of air to the cell restricted by rubber bungs in the open ends of the U-tube.

In the early stages of the test it was found that both the magnesium and the duralumin dissolved with practically no obvious evolution of gas or formation of insoluble corrosion product. The liquid in the anodic compartment (magnesium alloy) became mildly alkaline (*p*H 10·5, the *p*H of water in contact with magnesium) while that in the cathodic compartment became strongly alkaline (*p*H 13·0), and smelt strongly of ammonia.

On mixing the liquid from one compartment with the other, a thick white gelatinous precipitate was formed, containing both magnesia and alumina, while the supernatant liquid contained sodium aluminate. At this stage the magnesium alloy sheet had become noticeably thinner while the duralumin sheet consisted almost entirely of alumina, discoloured with copper oxide.

The reactions which had occurred, therefore, were evidently of some complexity and can perhaps best be expressed as follows :—

Anode Compartment—$Mg + 2NaNO_3 - 2e = Mg(NO_3)_2 + 2Na^+$

The sodium ion migrates to the cathode compartment where :—

Cathode Compartment—(i) $Al + 2H_2O + Na^+ = NaAlO_2 + 4H$; (ii) $NO_3 + 9H = NH_3 + 3H_2O$; and finally $Al + NaAlO_2 + 3H_2O = Al(OH)_3 + NaAlO_2 + 3H$.

This, then, is a somewhat unusual corrosion couple in that both anode and cathode are freely attacked, the attack on the cathode being due to the alkaline nature of the catholyte.

Several attempts were made to reduce the corrosion rate by anodising the duralumin, by the use of jointing compounds containing soluble chromates, or by the addition of sodium chromate to the fluid. The best results were obtained by the third method but they were not sufficiently good for practical application. Much better results were obtained by the use of a non-ionised de-hoar frosting liquid based on glycol.

It will be seen, therefore, that one of the most potent causes of the corrosion of magnesium alloys is contact with other metals in the presence of solutions of strong electrolytes. Magnesium alloys, therefore, should not be used under conditions where such contacts are likely to occur.

Damp Storage in the Presence of Wood

Another example of the failure of the normal methods used for the protection of magnesium alloys arose, in

* Translated from the original paper in *Métaux et Corrosion*, Vo. xxiv, No. 289, September, 1949.

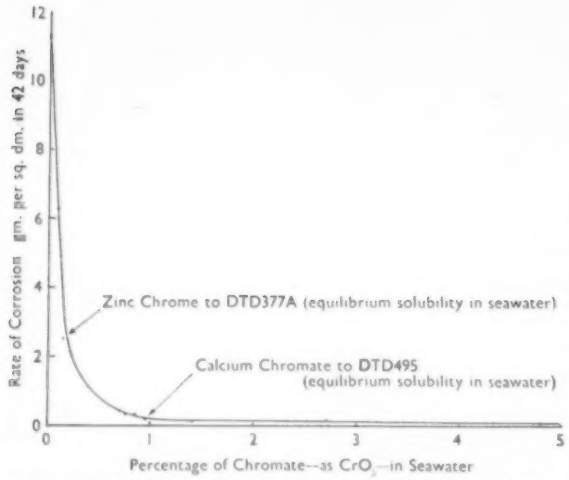


Fig. 1.—Inhibition by chromates of the corrosion of magnesium alloys by seawater.

which fully protected parts had been stored in a damp shed in poorly ventilated wooden packing cases lined with pitched paper.

The advice of the Forest Products Laboratory of the D.S.I.R. was sought, and it was suggested by them that the observed corrosion might be due to the emission of acetic acid from the wood, as it was known that such emission took place from various woods, particularly from oak and sweet chestnut, and that the acid produced caused serious corrosion of metals such as iron and lead.

To study the matter more closely, laboratory tests were made to simulate damp storage conditions with and without the presence of wood, pitched paper or the bakelised fabric insulating materials used in the electrical components attached to the parts. The tests showed that pure saturated water vapour alone had only a very slow action, even on unpainted chromate-treated magnesium alloy. In the presence of wood, however, corrosion took place quite readily. In the experiments in which pitched paper or bakelised fabric were used those materials became covered with substantial amounts of mould, in the presence of which some of the unpainted magnesium alloy specimens were attacked to give a markedly crystalline corrosion product which gave off carbon dioxide when treated with acid. The crystals appeared to be nesquihonite, $MgCO_3 \cdot 3H_2O$, and to result from a concentration of carbon dioxide, higher than that in the normal atmosphere, produced by the action of the fungus growth on organic materials. It was concluded that parts should be stored in open well-ventilated cases, pitched paper linings being on the whole detrimental rather than useful.

Later experiments on the wrapping of metal parts to protect them against tropical storage showed that, unless the sealing of the wrap was perfect and the contents dry to begin with, " breathing " took place and water condensed inside the wrap and remained there, a state of affairs worse than if there had been no wrapping at all.

Inhibitor Cartridges for Fuel Tanks

An important application of magnesium alloys in aircraft is their use in fuel tanks, in which magnesium manganese alloy D.T.D. 118 (A.M. 503) is commonly used on account of its easy weldability. It was early found that water separating from leaded fuel, which is rarely perfectly dry, is seriously corrosive to this alloy. The I.G. company had developed the " cartridge " method of corrosion inhibition, in which a small fabric bag of sodium or potassium fluoride is placed at the lowest point of the fuel tank where the water collects. It was found at the R.A.E. that zinc chrome to D.T.D. 377A and potassium chromate gave equally good results, and had the further advantage of being effective on aluminium alloys. Moreover, zinc chrome is only slightly soluble (approx. 0·07% CrO_3 when determined by the method described in D.T.D. 377A) and has therefore little tendency to be removed in solution when the tank is drained of water. Further study of the chemical properties of zinc chrome, however, showed that it acts by hydrolysis rather than by true solubility, and that the final product of hydrolysis is zinc tetroxy-chromate, closely similar in appearance to the original substance, but almost insoluble (0·0001% CrO_3). Inspection of the contents of an inhibitor cartridge by unskilled personnel would not show whether the cartridge was still active or not.

A search for a more suitable chromate led to the choice of calcium chromate which, though more soluble (approx. 1·0% CrO_3), is still sufficiently insoluble not to be washed out when water is drained from a tank. This salt, moreover, acts by true solubility and as long as any remains in the cartridge it is still active. The selection of this salt was also influenced by the results of experiments made for another purpose, in which magnesium alloy was immersed in seawater containing varying amounts of chromate. The results of these experiments, in which the weight of metal lost in a given time was plotted against the chromate concentration, are shown in Fig. 1. It will be seen from the curve that calcium chromate is better than zinc chrome to D.T.D. 377A, and also that little would be gained by using a chromate much more soluble than calcium chromate.

A disadvantage of calcium chromate, as normally supplied, is that it frequently contains considerable quantities of free lime which might be detrimental in applications where aluminium alloys are also present.

Fig. 2.—Corroded magnesium alloy petrol tank sump plate.

In searching for a method for overcoming this disadvantage it was found that, by adding milk of lime, with stirring, to chromic acid solution below 5° C., a solution containing as much as 28% of an unstable form of calcium chromate could be obtained.[1] This solution could readily be purified by adding a slight excess of lime, filtering, and bringing the *p*H value back to 10 by means of chromic acid. On boiling the solution, the normal slightly soluble form of calcium chromate was obtained as a sandy precipitate which was then drained off, quickly washed and dried. This material was found to be practically pure and had the advantage that, being gritty, it was much less likely to work its way through the fabric of the inhibitor bags than zinc chrome, which is a very fine powder.

Inhibitor bags containing calcium chromate of the above type are now giving very satisfactory results in service. An example of corrosion in a fuel tank where the above precautions had not been taken is shown in Fig. 2, which shows a magnesium alloy sump plate to which no inhibitor cartridge had been fitted and where no provision had been made for draining water away.

Corrosion of Welded Assemblies in A.Z.M. Alloy

During the war considerable trouble was caused by the corrosion of gas welds made in certain tubular assemblies of D.T.D. 348 (A.Z.M.) magnesium alloy which contained about 6% of aluminium. Welds made in this material are liable to be porous and it was found that, in spite of the normal cleaning and protective treatment, viz., scrubbing in water, immersion in boiling potassium dichromate for an hour, chromate treatment and two coats of paint, the assemblies were showing marked corrosion after a few weeks under rather indifferent storage conditions. Tests showed that the corrosion was due to welding flux entrapped in the rather porous seam.

A large number of experiments were made on different methods of cleaning, and it was found that immersion in a boiling solution of chromic acid followed by immersion in a cold hydrofluoric acid solution resulted in the removal of all the effects of the flux.

This procedure, however, was considered too complicated by the maker of the assemblies and further search indicated a very much simpler solution to the problem, which merely consisted of cleaning in the normal manner, baking at 120° C. and applying the undercoat paint to the structure while it was still hot. Though some flux was still entrapped by this method no corrosion occurred as no water was present in the interstices of the welds.

Acknowledgments

The authors wish to thank the Ministry of Supply for permission to publish the results of this work. No responsibility attaches to the Ministry for any expression of opinion in this paper, the opinions expressed being the responsibility of the authors.

Crown Copyright reserved. Reproduced with the permission of the Controller of His Majesty's Stationery Office.

1 LeBrocq and Cole, B.P.552685, Improvements in the Manufacture of Calcium Chromate.

Recent Developments in Refractories in The British Iron and Steel Industries

By W. A. Archibald, B.Sc., Ph.D.

THE maximum productivity of iron and steel, important in the past, is now absolutely vital, if a serious reduction of our standard of living is to be avoided in face of the increased requirements of these materials for rearmament. In the future, as in the past, much of the contribution which refractories' technology must make to the achievement of record outputs depends on progressive improvements in the quality and use of familiar materials. There are, however, some notable advances both in the iron and in the steel industries. Since there is no "perfect" refractory, these developments, evolutionary and revolutionary, have as their object the reduction of hindrances to production provided by limitations in the refractory—i.e., to allow higher rates of production uninterrupted for the longest possible periods at an economic cost.

In the iron blast furnace, for instance, the life of a lining normally of firebrick is sometimes brought to an early end by carbon disintegration, accompanied by slag attack and abrasion. This carbon disintegration, completely breaking down the structure, is caused by the deposition of carbon on the iron spots within the brick according to the reaction $2\ CO \rightleftharpoons 2\ CO_2 + C$ at about 500° C and is definitely the most spectacularly severe attack on the lining.

It has been avoided entirely in blast furnaces lined with brick made from china clay instead of fireclay. This result, which was a vast improvement in the plant concerned, was due to the freedom from nodular iron of the raw material, which showed similar behaviour to a good firebrick in slagging and abrasion. However, not only do both these types of alumino-silicate bricks interfere with production by requiring more frequent rebuilds, they also periodically contribute to scaffold formation because the slag sticking to them allows a basis for a build up. An ingenious solution to all these problems at once seems to be provided by carbon refractories. Both as a monolith and as large blocks keyed to prevent floating up, carbon has for some time been gaining acceptance as a hearth lining material. Trials culminating in a complete carbon lining have demonstrated that (a) it does not burn away (b) it is not of course subject to carbon monoxide disintegration (c) it has good abrasion resistance (d) it is not attacked by the slag (e) it does not encourage scaffold formation because the slag does not wet it. Further experiments will show to what extent it is possible to reduce the thickness and hence the cost of such a non-reacting lining. In certain circumstances the increased working diameter resulting may be an advantage as regards output. The

repercussions of this development on the chemistry of the process will take some time to be felt, as it offers freer control of the slag since attack on the refractory does not have to be taken into account.

In the steel industry the trend has been to seek improvements in the properties and use of silica bricks while exploiting more and more the properties of basic refractories. The introduction of metal-cased chemically-bonded bricks for use in the side walls of electric arc and open hearth furnaces is one development though the major activity has been in the extension of basic refractories to the roof of the open hearth furnace. The all-basic furnace, correct chemically for a basic process presents formidable structural difficulties. In the continental duplex processes where the metallurgical load and the temperature fluctuations are low the problem has been satisfactorily solved for some time, but here, where much cold metal is used, the acute temperature variations are the real snag. Even with bricks carefully made from raw materials giving adequate refractoriness and minimum tendency to expand by the absorption of iron oxide, a roof constructed in the same way as a normal silica roof would fail miserably. To realise the longest trouble-free life and allow the maximum increase in production a much more elaborate structure is essential. This is due to the fact that basic bricks have very different properties from silica bricks—

(1) They are heavier,
(2) The hot face tends to grow by iron oxide absorption and burst away on cooling even with the best raw materials,
(3) They have a much greater reversible thermal expansion than silica at high temperature,
(4) They are easily deformed and broken at high temperature.

Compared with silica a roof of these bricks therefore requires stronger external supports to carry the greater weight, restraining members to prevent it from buckling and some degree of suspension. The need for keying together provided by the usual sprung arch is necessary to prevent broken pieces from falling out. The expansion laterally is provided for by suspending the back skewback channel and holding in place by a number of helical springs.

The Continental basic roof designs began with the emphasis on restraining the roofs in shape, the American design with freely suspending the bricks; both have developed towards each other and both are now being given trials in this country.

The increased life of an all-basic furnace is prejudiced by weaknesses in parts other than the main roof and the present solution being adopted is the use of water cooling. This is used on some silica furnaces but is more essential on the longer-lived all-basic furnace, particularly on the skewback channel and the door arches and jambs.

The important contribution which operating conditions can make towards the life of the roof is in maintaining as steady a temperature as possible, mainly by keeping the fuel on during fettling and avoiding shut-downs.

Because of the more rapid melting down possible with the higher temperature available output increases of between 10 and 30% have been achieved and usually the limiting factor is not the furnace but charging facilities at checker conditions.

To date the evidence suggests that hard driving of the furnace is essential to make the more expensive refractory justify its cost.

In these trials the achievement of longer life and greater output economically has underlined the importance of the design and operating conditions and much work on aerodynamic models, on new ways of recording the refractory performance and studies of the conditions of temperature, stress, atmosphere, etc., are now being examined.

Problems in Powder Metallurgy

By H. W. Greenwood

Powder Metallurgy, Ltd.

Despite the progress made in powder metallurgical technique many problems have yet to be solved and they cover a big field. Probably the most important of them is the need for greater strength as measured by yield point and tensile, and greater resistance to high temperature. Promising solutions to these problems may result from investigations with combinations of metals and non-metallic refractories which are briefly discussed.

THERE are two directions in which greater and more rapid progress is wanted in powder metallurgy to-day and in part they are complementary. First, greater strength as measured by yield point and tensile, and second greater resistance to high temperature. It is obvious that the requirement to withstand stress at high temperatures is something very different from mere resistance to mechanical forces at room temperature. Indeed it is doubtful whether even now we fully realise the implications of the problem involved not only in the gas turbine as used to-day in aero engines, but in those which will be called for on land and marine practice as soon as they are available. Timely notice of the difference between aircraft and marine gas turbines was given recently in a leader in this Journal,[1] where it was pointed out that whereas the aircraft turbine is expected to have a life of 300–400 hours the marine gas turbine must be designed for a life of at least 100,000 hours, and no data is available as to the behaviour of metal when stressed for such periods at high temperature. It is worth noting before going any further that 100,000 hours is equal to 4,166 days of 24 hours each of a period of over 11 years. Little wonder that Dr. H. Roxbee Cox in his May Lecture to the Institute of Metals this year, said that the industrial and marine gas turbines were of more importance to the National economy than the more spectacular aircraft gas turbines and that metallurgists who have played so big a part in bringing the gas turbine safely through its infancy, have an even bigger part to play in guiding it, with due regard to economy, through its adolescence.

1 *Metallurgia*, June, 1950, page 1.

TABLE I

Stage	Elongation %	Tensile p.s.i.
(1) Press	nil.	150
(2) Sinter	4	15,000
(3) Coin	3	20,000
(4) Infiltrate	7	80,000
(5) 1st Heat treatment	14	60,000
(6) Straighten	—	—
(7) 2nd Heat treatment	8	90,000

Both the engineer and the metallurgist know that the efficiency of the gas turbine as an engine increases with the increase of the maximum permissible gas temperatures and, while methods of cooling may help by permitting the use of higher temperatures in the case of rotors and blades, these are not the only parts that demand resistance to stress at high temperatures. An example is recuperative heat exchangers where the present limit of temperature is of the order of 800° C. whereas a distinct advance upon this figure would be a great boon. It is in this connection that investigation of the cermets and their allies is of interest, for we most definitely have to look forward to the development of structural materials that can withstand stresses in the range of 980°–1,300° C. The hope has been expressed that "pure metallurgists" will take the work on cermets, and their like, as a challenge and it is likely that this will happen.

It is, however, important that at the moment we do not under-estimate the size of the problems that offer themselves for solution. Nor should we minimise the very big field they cover. In one sense we may divide them into two broad divisions, those dealing with increase of resistance to high temperatures without loss of too much strength, and those involved in increasing and controlling the physical properties of the materials we produce. It is not without significance that the last few years have seen really remarkable progress in both these directions, and in both cases it is the powder metallurgical technique that has made such progress possible. It may be emphasised that, in almost all cases of notable advances, the results have not been due to purely empirical methods, but in general to the application of information resulting from studies in pure science paralleling the more empirical attempts in the laboratory. As work proceeds there always arise theoretical questions that are capable of investigation and of providing, when studied, valuable basal data upon which to try out further experimental work. Examples of such interdependence are of frequent occurrence and some will be mentioned in the succeeding details.

Infiltrated Porous Sintered Bodies

Perhaps the most fruitful field in the last few years, in learning to increase and more accurately control the physical properties of certain metals and compounds, has been that of infiltrating sintered and porous bodies with another metal. The best known example of this is the infiltration of an iron skeleton with molten copper, so that not only are all the pores of the sintered skeleton filled, but there is a most dramatic change in the physical properties of the metal and various modifications in strength, ductility and other properties can be quite closely controlled within a most useful range. One of the most recent cases with reasonably full data was published recently.[2] In this case the product sought was compressor blades which, in the opinion of the authors, was "as challenging a problem as could be conceived for powder metallurgy." The part had previously been made by forging type 403 stainless steel. The blades operate at a temperature of 370° C., not an outside temperature as things are to-day. Experimental work had shown that an infiltration technique was a possibility and close study had shown what alloys were suitable, both for the sintered skeleton and for the infiltrant. The properties obtainable by suitable heat treatment had also been investigated and finally it was decided that the whole technique could and would solve the problem. A recital of the range of permissible properties will show that it was no mediocre result that was aimed at. For yield point the range had to be 65,000 to 90,000 p.s.i., while the tensile had to fall between 75,000 and 100,000 p.s.i., elongation must lie between 5 and 15% and Rockwell B hardness between 82 and 98. The process was carried out in seven stages and the progressive gain in physical properties, as well as the nature of each stage can be seen in Table I.

Infiltration was with copper, the sintered skeleton being iron, the first heat treatment finished with an oil quench, the second was a precipitation hardening. The tolerances maintained were width ± 0·008 in., thickness of airfoil ± 0·003 in., contour of airfoil ± 0·005 in. and, as will be seen, the physicals attained were almost exactly what were required. Up to the time the information was contributed over a million jet engine compressor blades had been produced within the required dimensional and physical properties. As to cost, it is said that the powder metallurgy produced blades were highly competitive with forged blades.

The fact that the compressor blade had an airfoil shape was, of itself, considered a big challenge on the basis of moulding alone, but there was the fact that a very unorthodox die had to be designed which had four lower punches, three for the airfoil, split longitudinally, and one for the root section. For the story of how this was done and of the difficulties met with and their final conquest reference must be made to the original paper.

The reason so much space has been given to this particular case is that here one has a typical example of the multiple problems set by almost any modern requirement in the metallurgical field. There is first the problem of material, whether simple or complex, and the general specification must determine the decision as to what material is used. Then there is how to attain the physicals that are called for and how to maintain the permitted tolerances. In this case the general requirement was a high strength part having close tolerances and capable of withstanding working temperatures of 370° C. Had it not been for recent experience and progress in the production of infiltrated material powder metallurgy could not have looked at the problem, because the very form of the airfoil with its thin, knife-like trailing edge could never have been produced from iron powder. It was to overcome this difficulty that the ingenious procedure of coining the iron pressing after sintering, but before infiltrating, was devised, followed by a heat treatment which gave hardness with the necessary strength and elongation. Such a statement as this fails to take into account many other special problems that had to be solved as experimental work proceeded, but it does give some idea of the close-knit group of major problems that had to be solved, and of how the whole design and execution of the work demanded the

[2] *The Iron Age*, February 23rd, 1950, pp. 74–77.

closest integration and study at every stage of the process. Inasmuch as the final requirements were not exceptional the case suggests something of the complexity of the further problems involved in producing gas turbine blades for air and marine service which can stand up to temperatures in the range 900° C. and upward. High creep rates and low resistance to oxidation are outstanding difficulties to-day, even with temperatures of about 820° C. (1,500° F.), and provide materials of comparatively short life.

It is of the utmost importance to remember that for land and marine service turbines will not be using as fuel, aviation petrol, but cheaper fuels of distillation residue types and hence resistance to oxidation and also to oxidation accompanied by abrasion by ash from the fuel used will be increasingly essential. It is already known that many residual fuels carry an appreciable vanadium content and that this has the effect of lowering the melting point of the ash to about 650° C. As marine and gas turbines to-day have a gas inlet temperature of 600°–700° C. the ash comes into the turbine in a molten or sticky state and provides a source of first-class trouble as it attacks all the well-known steels and alloys used for turbine blades, etc. Hence the importance given to the possibility of liquid or air cooling which offers elimination of corrosion and possibly reduction of blade fouling. A drawback here is the fact that a porosity of such an order as to permit of circulation either of air or liquid adequate for efficient cooling would probably lower the physical strength and properties to such an extent as to eliminate it from practical use.

The Cermets

Another line of attack on the problem is in the use of combinations of metals and non-metallic refractories, cermets as they have been called. This is an attractive line of investigation which already offers promising results. The technique follows reasonably closely the methods of powder metallurgy in that the raw materials in powder form are mixed, pressed and sintered. Generally speaking combinations of metal such as chromium or iron with alumina or with one of the new refractory bodies such as boron carbide or zirconium boride will have densities, strength at high temperatures, oxidation resistance, and resistance to thermal shock, falling between the properties of the individual constituents. Moreover a large measure of control is possible by varying the ratio of the constituents. Alumina is probably one of the best known ceramics. It has a tensile of about 38,000 p.s.i. (17 tons) at room temperature which falls slowly up to nearly 1,000° C. and is still 20,000 p.s.i. at 1,200° C. Addition of a metal such as iron reduces the resistance to high temperature. With alumina and iron proportions used are usually between 40 and 50% iron. A general rule is that metals having oxides of the same crystal form and lattice parameters as the ceramic oxides and forming a continuous series of solid solutions with them are the most suitable. One of the successful cermets to date is Metamic LTI produced by Haynes Stellite which contains 70% chromium and 30% alumina. It has good strength up to about 1,000° C., high resistance to oxidation in air up to 1,200° C. and to gases of combustion up to 1,600° C., resists attack by molten steel and yet can be copper brazed to steel.

The cermets offer many possibilities for, as yet, no great volume of work on them has been published. Another field with outstanding potentialities is that of cemented carbides. Generally we think of carbides cemented with a fair proportion of a metal of the iron group which acts as a liquid phase during sintering. There are two other alternatives to such a procedure which offer interesting possibilities. One is the use of hot pressing, especially for the production of difficult shapes and forms. The other is a recently introduced modification by which the lower melting point cement is distilled off leaving a porous mass of carbide with little or no low melting point constituent. Up to the present, so far as is known, this technique has been used with tungsten, columbium and tantalum carbides to provide zero surface-connected porosity and approximately theoretical density, but there is no reason to restrict the technique to such production. In the details published very finely ground components were used and very small amounts of cementing material, in order to obtain the specific result. There are possibilities of extending this technique and using a carbide or other refractory skeleton as a basis for other combinations.

The much greater knowledge and experience in handling refractory metals we possess to-day should be of service here and the fact that platinum (M.P. 1,773° C.) has already been used with success as a cement for carbides, in cases where chemical attack on cobalt or nickel had to be met, suggests that in other directions we should be able to think of infiltrants such as chromium (M.P. 1,615° C.), titanium (M.P. 1,800° C.), or zirconium (M.P. 1,900° C.). In addition to their refractory qualities and their hardness, titanium and zirconium offer advantage because of their low density and their resistance to oxidation. In this they occupy a position of greater importance than molybdenum and tungsten whose oxides are definitely volatile at temperatures over 1,200° C. This leads to rapid oxidation of these metals due to the successive presentation of fresh surfaces to oxidising influences as the already formed coat of oxide is removed by volatilisation.

Although they have not been mentioned specifically other refractory compounds are not to be overlooked and materials such as borides, nitrides and compounds of silicon have served as experimental materials. In all cases the most promising approach to such problems as exist and the basic technique is by powder metallurgy for it is by powder metallurgy that the broadest fields may be explored and combinations experimented with which are not amenable to general metallurgical processes.

Alumina Production in Jamaica

Plans for the development of the Jamaican bauxite deposits have been under consideration since 1942, just after their discovery, as a supplementary and strategic source of supply for the large Canadian aluminium smelters. Emergency running plans did not materialise during the war, but after study of the special nature of Jamaican ores, and the economies of their ocean transportation it has been decided to undertake not merely bauxite mining in Jamaica, but also extraction of alumina from bauxite through the construction of a plant capable of producing 100 tons of alumina per day. If the process is successful, and market conditions warrant, operations could be expanded tenfold with an additional investment of up to $30m. Large port works would then be required.

The plant will be built and operated by Aluminium Limited's fully owned subsidiary, Jamaica Bauxites Ltd., whose contract with E.C.A. was announced in Washington and London last month.

Co-operative Research Activities

Reports of Research and Development by British Associations

The gulf that formerly existed between scientific knowledge and its application is being narrowed and to-day it is more fully recognised that progress is dependent upon the ability to use this knowledge. This is particularly true in industry where the application of research is doing much to improve and quicken production. Contributing to this progress are many co-operative research organisations, which, for many years, have been functioning with increasing success, and the following reports will keep readers informed of the activities of those within our field.

WITH the increasing need for armaments and men for the Forces there will be greater strain on Britain to balance her imports of food and raw materials with exports of manufactured goods, without reducing the standard of living. Since she must buy from overseas it is more than ever important that her products exported are competitive in quality and cost. This involves constant research and development and the rapid application of the results, not only to improve products but also to achieve greater productivity and thereby reduce costs.

Research leads to the development of new processes, or improvements in existing processes, new machines, new materials and new applications for these and existing materials, and, under modern conditions, an industry can only thrive and develop when its production facilities and materials are being continually examined with a view to effecting improvements; it must be keenly alive to progress in its particular field. Many firms have their own laboratories where investigations are carried out with a view to improved and cheaper production, where new products and more advanced production techniques can be developed and where production problems can be carefully examined and solved. Other firms without these facilities were at one time at a serious disadvantage. For many years, however, co-operative research organisations, partially supported by the Government, have been carrying out research and development work in particular fields of activity and, whether a firm has its own laboratory or not, membership of the appropriate organisation entitles it to service in the solution of its production problems. It is in connection with the work of some of these organisations that the following reports are concerned.

The British Iron and Steel Research Association

Some Development Work in Progress is Reported

This Association is organised under six Divisions and at any one time it is engaged on some 350 projects, covering the whole industry from raw materials to finished products. In this report, therefore, it is only possible to refer briefly to some of the work in progress.

THE work of the British Iron and Steel Research Association is organised under six Divisions, which are mainly concerned in their respective sections of the industry with applied researches. These divisions are aided by two departments concerned with more fundamental work, respectively in the fields of Physics and Chemistry, the latter mainly physical chemistry. Each of the divisions has some laboratory space, and considerable extension is in progress at the Association's research station in Sheffield, where much of the work which can best be carried out on semi-scale plant is being concentrated.

The two departments have their laboratories in Battersea, London. This month sees the third anniversary of the opening of these laboratories, and this report therefore starts with a note on part of the work carried out there.

The Physics Department has played a major part in establishing the international Joint Flame Radiation Research project at the Royal Dutch Steelworks in Ijmuiden, Holland. Here workers from France, Holland, Sweden and Great Britain are elucidating some of the problems of flame radiation, which are of importance to all engineers concerned with heat transfer from flames, as in boilers, gas turbines and furnaces. A series of 47 factorially planned engineering experiments has been carried out, in which each of 5 variables was given 2 values. The work now in progress is the more detailed investigation of conditions within the flame.

Another major project of the Physics Dept., is the study of the physical problems which make the continuous casting of steel so much more difficult than that of non-ferrous metals, which is already in commercial use. The stage has now been reached at which a specially designed apparatus gives a comparatively slag-free vertical stream of steel, directed into a water-cooled mould. Sticking, which has been the cause of failure of many continuous, casting processes, is now largely eliminated by the technique which has been evolved. Latterly, work has been on 18/8 stainless steel, and it is now possible to cast a complete crucible charge of 50 lb. at a speed of about 4 ft. per minute to give bars 2¼ in. in diameter and 4 ft. long. The surface quality of these bars, while showing a considerable

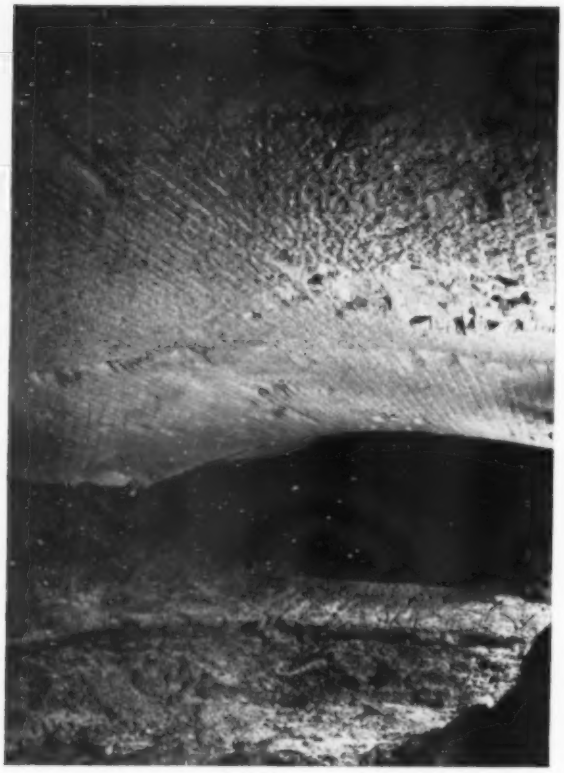


Fig. 1.—Showing the single air uptake in the new design of open hearth furnace.

improvement over the shorter lengths cast earlier in the year, is still unsatisfactory, and is the subject of continuing study and experiment.

Part of the work of the Chemistry Department is concerned with the thermodynamics of phosphorus in liquid iron, with the object of determining the concentrations of phosphorus which can exist in liquid iron in contact with an idealised slag, in this case pure lime and tetra-calcium phosphate, under various oxidising and reducing conditions. Another project is concerned with the mechanism of the reduction of wüstite, which was originally stimulated by the lack of accurate data on the kinetics of reduction of iron oxides. In order to obtain a reproducible sample, a thin film of oxide is formed on pure iron foil and the speed and mechanism of its reduction in hydrogen observed.

Both the Physics and Chemistry Departments have, of course, many other projects in hand.

At any one time BISRA as a whole is engaged on some 350 research projects, covering the whole industry from raw materials to finished products and their most economical use. Any notes on the work of the six divisions must therefore be highly selective.

Ironmaking

An important part of the work of the Ironmaking Division is concerned with the preparation of raw materials, and the assessment of the effects of changing conditions or sources of supply. BISRA observers have been present, for example, at three works when full-scale blast furnace trials were carried out on cargoes of Konakry ore from French Guinea, these being the first received from new workings. Data have been provided for metal and slag analyses (in conjunction with the Bragg Laboratory) and top gas analyses. Statistical examination of the data is also being made, in which the Physics Department again plays an important role. In general, the trials showed that in spite of high alumina, the ore can be worked satisfactorily, provided that other burden constituents are available in proper balance. These trials have been carried out at the request of the British Iron and Steel Corporation (Ore) Ltd., the industry's central ore buying agency, for whom such information is clearly essential.

Interesting experiments with considerable possibilities are being carried out on a blast furnace at the works of a member firm, where lime is being injected at the tuyeres. Quantities up to 3 tons have been introduced in 2½ hours at half the tuyeres without significant change in lime content of the flue-dust or detriment to the hearth. Slag analyses show a time delay of 1–2 hours before the maximum lime content becomes apparent. This compares with a time delay of perhaps 7 or 8 hours with the normal feeding operation, and promises a speedier method of slag control by that margin.

Steelmaking

An excellent example of co-operative research at work is given by this division's work on the "single uptake" open hearth furnace. Works are reporting increased outputs of up to 20% since the installation of this simplified design of air inlet or "port" co-operatively developed by the industry, though the usual reservation should be made that steelmaking is a complicated operation, and major results such as these are seldom the consequence of one factor alone. In this new design a single air uptake or inlet is substituted for the usual two. It is placed at the end of the floor of the furnace, and centrally under the gas or oil inlet, blowing the air upwards nearly at right angles to the incoming fuel. In the more usual designs the air for combustion is moving almost horizontally as it meets the fuel stream. The result of the new arrangement is improved mixing, with quicker, more efficient combustion.

Such a major change in design could not be made without exhaustive and often tedious investigation. This was particularly important in this instance because of the danger of deflecting the flame upwards, on to the furnace roof. Trials on B.I.S.R.A.'s experimental furnace in the works of a member firm began in 1944, while at the laboratories of another member firm and in B.I.S.R.A.'s own laboratories gas flow patterns were investigated on models by various techniques. In due course trials were extended to production furnaces, and in particular to one in the melting shop of the manager who had first advanced the design for B.I.S.R.A.'s consideration. By mid-1949 enough experience had been gained for the new design to be made the subject of a conference of steelworks technicians and scientists. The advantages were described as simpler construction, higher outputs, longer furnace life and better sulphur removal, though not all these could be otained at once.

The development of oxygen lancing in steel manufacture in electric arc furnaces has been another project of this division. A description of its use in two of the several steel works employing it was given in METALLURGIA for June, 1950.

Steel Castings

Two projects in this field may be touched upon: hot tearing and burning-on. The hot tearing of castings, where fractures occur as the result of cooling stresses, is a long-standing problem. Research has now shown that a tearing temperature is generally of the order of 1,400° C. and that a reduction in the casting temperature reduces the temperature of tearing and increases the stress required to cause it. The precise effects of various alloying elements is becoming apparent, and it appears that carbon above 0·2% and most normal alloying elements added in small quantities increase susceptibility to tearing, but further additions of chromium, molybdenum, titanium and vanadium reduce this tendency. Already it is possible to exercise better control over hot tearing and, gradually a picture is emerging which will enable that control to be made more precise.

Burning-on, the firm adherence of sand to the surfaces of castings is another long-standing problem now yielding to treatment. Continued laboratory studies have shown that metal penetration of the mould does not take place progressively, but rapidly on the attainment of specific conditions and then remains reasonably constant; casting temperature, metal fluidity and mould permeability being major factors in governing this condition. It has been found that penetration occurred in all cases where the temperature at the mould face immediately after pouring exceeded the solidus temperature with uncoated moulds. Here, again, increased knowledge is enabling control to be improved.

Mechanical Working

The Mechanical Working Division is concerned with almost everything that may happen to steel from the ingot to the finished product, including rolling, forging, pressing, drawing, coating and all the many problems these processes bring in their train. In rolling steel strip, for example, it is extremely difficult to maintain a uniform gauge throughout the length of a coil. With the increase of the continuous rolled strip process for the production of sheet and tinplate this problem grows rapidly in importance. A common procedure is to observe the readings of a flying micrometer and to adjust the screwdown gear as required. A new method which has recently been tested on the B.I.S.R.A. experimental mill is to observe the readings of a roll force meter while rolling with a fixed screwdown setting and to adjust the strip tension to keep the roll force as constant as possible. Very uniform strip is produced in this way and work is in hand, in collaboration with the Plant Engineering Division, to make the process fully automatic. The obvious advantage is that the delay attendant on adjusting the screwdown is eliminated.

Another major problem the steel industry has to face is the need for economy in the use of sulphuric acid for pickling, particularly important in the making of continuous strip. The "spent" pickling acid is a solution of ferrous sulphate or copperas, into which the sulphuric acid used for descaling steel has been converted during the process. A method of regenerating fresh sulphuric acid from the copperas has been developed at B.I.S.R.A.'s South Wales laboratory and plans for semi-scale operation at a steelworks in South Wales are well advanced. The copperas, mixed with coke, will be flash roasted and the oxides of sulphur produced will be absorbed directly in water and oxidised catalytically, giving a sulphuric acid of strength suitable for pickling without further dilution. An economic appraisal of the process operating on a large scale indicates that it should result in substantial savings to the industry.

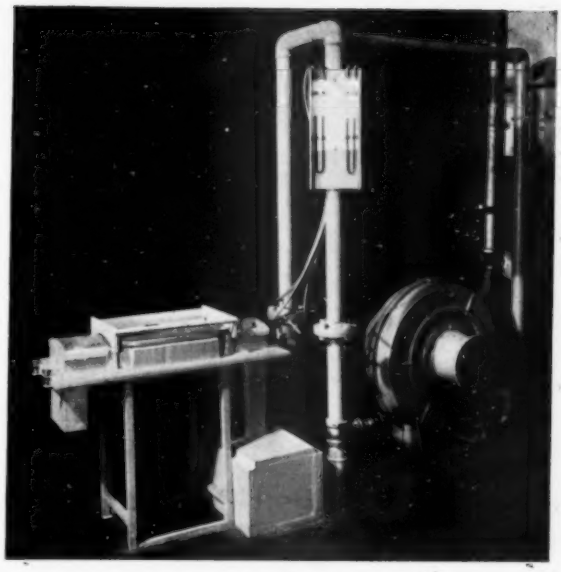


Fig. 2.—Model of oil-fired open hearth furnace, together with blower, used to determine general aerodynamics of the furnace including dust disposition.

Plant Engineering

Only the electrical engineering section of this division will be touched on in these notes, space forbids a description of the active work on which the civil and mechanical engineering sections are engaged.

Since May, 1949 the Plant Engineering Division has had a small laboratory at Battersea which is occupied by the Electrical Engineering Section. The work carried out here has been mainly concerned with the development of a new equipment for performing multiple screwdown adjustments, while the hot strip is in the mill, with the object of improving uniformity in longitudinal gauge. The control unit is being arranged to follow automatically a pre-set programme determined from measurements taken on production strips. The control scheme is being developed at a much lower power level than is usual in steelworks plant, by employing standard post office telephone equipment. This gives a great latitude in circuit design and substantial saving in cost and bulk of equipment.

Another project concerned with the control of longitudinal gauge of cold strip by adjustment of roll force which, in turn, is dependent on strip tension, has been mentioned above. Plant Engineering's part in the project consists of devising a closed loop control and in assessing its stability both in practice and theory. From this it is hoped ultimately to produce a simple and stable control system acceptable to the industry.

Crane cab and pulpit controls are also receiving attention. Several master controllers radically improved in shape are under construction, or under test in steelworks and when these have been proved they will be incorporated in crane cabs and pulpits designed on

correct economic principles. Very great improvements will undoubtedly be possible as a result of this investigation.

An investigation into the suitability of the available British VHF radio equipment, for use in communication systems between traffic control points and steelworks locomotives is nearly completed. Tests to date confirm B.I.S.R.A.'s original contention that low powers of 3–5 watts are adequate for most sites.

A survey has been made of the opinions of engineers on the subject of mill motor rating and their application to steelworks auxiliary drives. This has confirmed a lack of knowledge on duty-cycles and the frictional load characteristics of auxiliary drives. As an initial approach to the ultimate aim of improving the system of rating mill-type motors to facilitate application, tests will be carried out in steelworks to obtain data on duty-cycles and motor loading.

Metallurgy Division

This division is concerned with the general and physical metallurgy of iron and steel, including special steels.

One important series of projects is jointly financed by this Division and the British Electrical and Allied Industries Research Association, and is concerned with steels for high temperature service. It includes research at the National Physical Laboratory on the assessment of the effect of various metallurgical factors, including composition and heat treatment, on the creep properties of a number of steels. The investigations of the causes of cracking in steam pipes, the effect of cold drawing on the properties of molybdenum steel pipes, and the effect of various commercial treatments on the properties of carbon steel tubes have also progressed.

New work on the development of steels to resist even higher operating temperatures in steam plant includes the development of new ferritic steels for superheater tubes, and the creep testing of specimens from actual rotors and pipes.

Work on methods of analysis provides another example of co-operative research, of importance to steel makers and users alike. Some of the most important developments in ferrous metallurgy during recent years have been obtained by small, controlled variations in composition. Reliable and reproducible determinations of alloying additions can only be obtained by using perfected and standardised analytical methods. The Methods of Analysis Committee has the responsibility of providing and recommending such methods, and maintains close liaison with the British Standard Institution. Approved standard methods are published as parts of B.S. 1121, additions over the past year having covered the determination of copper, maganese and chromium in steel. Tentative methods for tin in alloy steel and ferro-tungsten, and for chromium in ferro-chromium have been completed and reports have been published on the investigations undertaken during the development of the methods. Since the Committee's inception in 1946, sixteen methods have been published in B.S. 1121.

Corrosion

The work of the Corrosion Committee and its Sub-Committees comes under the Metallurgy Division but is technically supervised by the Chemistry Department. A report has recently been published on extensive tests carried out by a joint technical panel on marine corrosion, designed to study the effect of changes in the medium and the influence of small changes in pigmentation on the protective properties of two of the best compositions from earlier experimental work, which (coded as formulations Nos. 173 and 185) have found wide acceptance in both naval and mercantile practice.

Eight of the 68 compositions tested are placed in the "very good" group, all of which appear to be improvements even upon earlier formulations. Paints bound in chlorinated rubber were among those which gave the best performances, thus confirming earlier raft tests at Caernarvon. They are closely followed by a phenol-formaldehyde/stand oil/tung oil medium.

The effect of pigmentation was slight, but the tendency was for an increase in the aluminium content to result in improved performance, as regards both protective properties and can stability. Three compositions in which Burntisland red was replaced by a natural red oxide gave better results than the compositions from which they were derived, but further work is required before advocating this change.

From considerations of performance and general applicability the most promising composition studied in this programme is composition No. 655. This has a pigmentation of two parts of basic lead sulphate with one part of each of aluminium powder, barytes and Burntisland red bound in a modified phenol-formaldehyde/stand oil/tung oil (ratio of oils, 1 : 1) medium.

Galvanizers Visit U.S.A.

UNDER the auspices of the Anglo-American Council on Productivity, a specialist team on galvanizing left for the U.S.A. on September 14, and is due to return from New York at the end of October.

Because galvanizing is not really a separate industry in this country, but rather one process in a large number of industries, the main need is a survey of galvanizing techniques in America. It is for this reason that the team of fifteen, under the leadership of Mr. Frederick C. Braby, chairman of the Hot Dip Galvanizers Association, consists largely of specialists, in contrast to the usual teams comprising representatives of management, technicians and operatives.

The American Hot Dip Galvanizers Association is taking great interest in the visit and the plants of several of its members will be inspected, as well as other plants, so that the covering of processes and of works of various sizes may be fully representative. Hand and mechanical dipping and the continuous process will be studied and particular attention will be paid to general research on galvanising and to research on the corrosion of galvanised coatings.

The tour, which will cover a wide area of the eastern half of the United States, will be a strenuous one. In addition, members of the team have been invited to attend the convention of the American Hot Dip Galvanizers Association at White Sulphur Springs, Virginia, where a paper will be read on behalf of the team.

The Sales and Administration Offices of the Stella Lamp Co. have moved, on July 31, 1950, from their offices at Winchelsea Road, Harlesden to :—37-39, Oxford Street, London, W.1. Tel: GER. 8315. Telegraphic Address: Stelamp Wesdo London.

The British Cast Iron Research Association

By Dr. J. G. Pearce, O.B.E.
Director of the Association.

Although new buildings and extensions to existing buildings, and their equipment to enable the Association to serve its members are emphasised in this Report, they clearly indicate the increasing demands on the services of the Association which is substantially reducing the gulf that formerly existed between technical developments and their application in the founding of cast iron.

External view of the new building housing the Scottish Laboratories.

SEVERAL notable developments have taken place during the past year. On May 5th Lord Bilsland opened the new Scottish Laboratories of the Association at Blantyre, Glasgow. These were centred at Falkirk for over 20 years. Over 5,000 sq. ft. of covered space are available in six laboratories, with auxiliary accommodation, and an external view is shown in Fig. 1. On July 5th, Mr. P. H. Wilson, O.B.E., M.I.Mech.E., President of the Association, opened the extensions at headquarters, Alvechurch, Birmingham, in the presence of some 200 representatives of members. The new buildings and equipment were fully described in the July, 1950 issue of METALLURGIA, page 91. The total covered area at Alvechurch is now some 20,000 sq. ft. and the capital assets in buildings and equipment approach £100,000.

During the year a decision was taken to set up an Operational Research Team to visit member-firms and to assist them by means of confidential reports to promote technical and productive efficiency. Two of three appointments envisaged to the team have been made and visits have begun.

The Association has always been closely concerned with foundry working conditions and in particular with the technical aspects of measures required to implement the 1947 report on foundry working conditions of the Joint Advisory Committee of the Ministry of Labour and National Service, the so-called Garrett Report. In close collaboration with the Factory Department of the Ministry, some work was begun at Loughborough College, sustained by the Association, on the nature of products from corebinders after baking. On account of staffing problems, this investigation has regretfully been abandoned. Other measures are contemplated and a decision has been reached to appoint a suitably qualified engineer to provide an advisory service to foundries. As all iron foundries in the U.K. may become members by signing an application form, this and other services provided by the Association are available to all member-firms.

The research programme has proceeded normally and since mid-1949 no less than 33 reports have been issued in the Association's Journal of Research and Development, nearly double the number in the previous year. This publication has been made available by the Council to non-members on a subscription basis.

The Association has held two conferences at Ashorne Hill—in September, 1949 on foundry sands, and a conference on general foundry subjects in March, 1950. On both occasions the accommodation was filled to capacity. The number of enquiries handled by the Intelligence Department and Library (about 2 per working day) and by the Development Department (about 4–5 per working day) indicate a very close connection between the Association and its member-firms. The number of Library loans has reached a record figure of about 20 per working day.

Two members of the staff of the Association, Dr. H. T. Angus, Development Department Manager, and Mr. W. B. Parkes, Assistant Manager, formed part of the General Ironfounders Productivity Team which visited the United States in January. This was the first iron foundry team to pay such a visit and their report is approaching completion.

The Scottish Laboratories

Previously housed in a building rented from Stirlingshire Education Authority and located in Falkirk the Scottish Laboratories were originally formed by the National Light Castings Association—now the British Ironfounders Association—under the name of the Foundry Technical Institute. They were opened in December, 1918 under the guidance of a Committee of Management and with Mr. A. Campion, F.I.C., F.C.S., as Superintendent and Metallurgist. In 1925, the British Cast Iron Research Association took over these Labora-

tories in Falkirk and the membership in Scotland was opened to all classes of ironfounders. A steady increase in membership followed and in 1944 the Laboratories were completely remodelled. The introduction of the Joint Iron Council, scheme, in 1948, resulted in a further increase in membership and the need arose for larger premises to accommodate the increasing demands made on the Laboratories. The outcome was the acquisition of the premises recently opened.

The main functions of these Laboratories are to receive and deal with inquiries and problems from Scottish and Northern Counties' members and to aid members in the application in their own foundries of scientific data from the Association and elsewhere. The premises chosen for the new laboratories, a recently erected 5,000 sq. ft. unit in one of the standard estate blocks, have been adapted to meet these special requirements by substantial internal alterations and additional building. In addition to clerical and administrative offices, the building includes the chemical laboratory, balance and furnace room; Spekker laboratory; metallographic laboratory, with adjacent dark rooms; machine shop; mechanical testing laboratory; sand, fuels, and refractories laboratory; and development laboratories. Equipment and apparatus are installed for carrying out the work in the various sections and the laboratories are adequately staffed.

Laboratory Extensions at Headquarters

The extensions at Bordesley Hall, Alvechurch, previously mentioned, comprise a machine and instrument shop for the preparation of test pieces and the construction of experimental equipment; a mechanical testing laboratory; and a staff dining room. The building, formerly occupied by the machine shop, is being taken over by the chemical laboratory and an adjoining room will accommodate the chemical research section.

The British Welding Research Association

Notable Features of the Year's Work

Work on various researches has been substantially increased during the year under review. Particular attention is directed to work on the high temperature properties of weld metal; the weldability of two selected steels; the corrosion of welded gas mains; the examination of pipe lines, and the welding of light alloys.

WITH the advent of new machinery and additional Ministry of Supply contracts, work on various researches has been increased considerably during the year under review, and it is to the credit of the staff that so much has been accomplished in the very limited laboratory accommodation available, both at the London metallurgical laboratories and at the Research Station, Abington. Plans have now been made, however, for a fatigue testing laboratory to be constructed at Abington with a floor space of some 5,000 sq. ft. which will do much to lessen the present crowded conditions, and which will also house the new vertical fatigue testing machine which is on order. This machine is eminently suitable for carrying out static tensile, compression, buckling and bending tests up to a maximum load of 200 tons, and for determining the fatigue strength of materials and structural assemblies up to a maximum load of 100 tons tension or compression. With such a machine, of which there is only one of similar capacity in the country, much valuable work can be carried out both in regard to economy in the use of steel and in design.

With the same objects in mind a pulsating pressure plant has been installed which is the first of its kind in the country, the pressure range being between any two limits up to a maximum of 6,000 lb. per sq. in. It is considered that the Association now possesses a unique laboratory for testing pressure vessels and pipelines and future researches carried out should accordingly be invaluable to British engineering.

With a view to displaying various aspects of the Association's work to the engineering industry and to emphasise the link between research and production an exhibition was organised at the London offices and subsequently in Glasgow. This was extremely well attended at both places and considerably widened the interest of industry in the work of the Association.

The liaison and development department was expanded in August, 1949, and has already proved its worth; between August, 1949 and March, 1950 alone, some 230 engineering companies were visited and some 500 queries dealt with.

Notable features of the year's researches include investigations into the high temperature properties of weld metal; the results have been presented in a series of reports which will shortly be incorporated in one document for circulation to members. The causes of halo formation in the weld metal are still under investigation.

Weldability of Selected Steels

Two-ton casts have now been made of selected steels for investigating weldability; the two selected steels have been rolled into plate of ½ in., 1 in. and 1½ in. thicknesses and it has been found that the larger casts did not have mechanical properties as high as those obtained with steels of similar composition from the small experimental melts—probably due to the slower cooling rates obtained with the thicker plates. Work on the weldability of a particular low alloy high tensile steel has continued during the year and has resulted in a weldability test of variable severity.

Corrosion of Welded Gas Mains

The research on the corrosion of welded gas mains is still in progress and arrangements are now well in hand for the installation of a full size experimental gas main at Doncaster, in which different types of steel and various methods and degrees of stress relief after welding are to be investigated.

Frame Structures

With regard to the work on load carrying capacity of frame structures, considerable progress has been made in applying the simple plastic theory to the design

of rectangular and pitched roof portal frames and in comparisons made an indication has been given of the extent to which the normal variation in properties of rolled steel to B.S. 15 are likely to influence the behaviour of rigid frame structures when loaded into the plastic range. A number of tests have been carried out on small I-beams to investigate the lateral stability of structural members loaded beyond the yield point of the material. Using the same section throughout (2 in. × 0·86 in. with $\frac{1}{8}$ in. flanges and $\frac{1}{16}$ in. web, a scale model of a 10 in. × 4½ in. joist) tests were made on several lengths for which instability occurs in the plastic range. In each case yielding occurred in the compression flange before buckling took place; tests on two considerably longer lengths were also carried out, both of which buckled in the elastic range. The critical unsupported lengths for the British Standard Rolled Beam Sections have also been calculated. These small scale members have at present been subjected only to pure bending; hydraulic loading gear has now been constructed in order to apply transverse loads. The third full-scale portal frame has been tested and the results obtained from the whole series is now proceeding and a full report is in course of preparation. Work has continued on the application of the plastic theory to the design of practical portal frames; a number of types of single bay frames have been examined, including unsymmetrical frames and those carrying heavy overhead gantry cranes. The British Constructional Steelwork Association has appointed a special panel to consider the plastic design of portal frames and the basic work for a handbook for the instruction of draughtsmen is being carried out under the aegis of the Association. Investigations have also continued on the fatigue strength of welded joints in angle stiffeners attached to ship plating.

Radiological Examination of Pipe Lines

In work on welded pressure pipe lines particular attention has been given to radiological examination and the technical and economic factors which influence its use in this class of work; the subject has been considered and in a memorandum issued to members attention has been drawn to the different views held on the necessity for specifying radiological examination. First class results have been obtained with the extensive use of electrical resistance gauges and the development of scanning and recording equipment, in investigating stresses in welded pressure vessels. Pressure tests have been carried out on two 6 in. diameter 90° pipe bends to investigate the behaviour of seamless and welded bends; a portion of straight pipe having a similar bore and wall thickness was also tested under internal pressure to obtain data on the bursting pressure and the occurrence of yielding. The pressure at failure was 6,650 lb./sq. in. Electric resistance strain gauges were applied to the inside and outside surfaces and measurements carried out up to 5,000 lb./sq. in. all gauges remaining fully waterproofed up to the highest pressure encountered in this test. Investigations have also been commenced on curved ducts having extension tangents welded on.

A series of statements has been collected on various non-destructive methods for examination of welds and these are being examined in committee and prepared for issue as a single authoritative statement on this important subject.

Welding Light Alloys

In the work on light alloys, a technique for the pressure welding of various metals in sheet form (18 gauge) is being established; the optimum conditions for cold welding have been found, with particular attention being given to prior heat treatment. Work has also been continued in the fusion welding of aluminium and magnesium-rich alloys.

Summary of Progress

The year under review marks the end of three years' extensive research on the projection welding of mild steel to Specification S. 84, and various reports thereon have been issued; as a result of a statistical analysis of the results it was found that better values of consistency were obtained with single projection weld specimens than with multiple projection welds and further exploratory tests on these latter welds are being made. The work will eventually form the basis of a Technical Memorandum on Projection Welding. The resistance stud welding process is also receiving attention with a view to elucidating optimum conditions for making satisfactory welds; particular interest attaches to the form and size of stud and for welding sheet steel and so far experiments have been made with radiused heads which are simple to produce commercially by cold working; it is hoped that the work will lead to a standardised form of stud preparation for this process. A Technical Memorandum for the spot welding of mild steel sheet in the range of 20 to 14 s.w.g. (confined to mild steel of specification S.84) has been published and tests are now in progress on certain thicknesses of mild steel above and below these limits.

Reports on the resistance welding of light alloys based on the past year's work will shortly be available for Members of the B.W.R.A. and include "Surface Preparation" and "Machine Instrumentation"; reports already issued are "A Review of Selected Papers on the flash and butt welding of light alloys" and "A Review of recently published information on the spot welding of light alloys."

Membership of the Association has increased during the year and there were at 31st March, 1950, some 197 members and 11 associate members. To serve these the library is being improved and a start has been made on the formation of a films library.

Service Van for Welding Machines

A NOTABLE move of British Insulated Callender's Cables Ltd. to encourage closer relationship between user and manufacturer is the establishment of a mobile servicing unit for their resistance welding machines. The unit will be in the charge of a specially trained service engineer, and will consist of a 10 cwt. van fitted to carry tools, spare parts and welding accessories. The service engineer's duties will comprise servicing of BICC machines, advising customers on their welding problems, and the selling of Bicaloy tips, electrodes and other welding accessories. No charge is made for the service inspection, other than the cost of accessories supplied.

In addition to an efficient service being maintained the scheme will increase customers' confidence in the welding machines used, and although at present only one van is employed, the scheme is likely to be extended after the initial trial period.

The National Physical Laboratory

Fundamental Metallurgical Research in Progress

By N. P. Allen, D.Sc., M.Met.
Superintendent of the Metallurgy Division

The need for giving priority to short term problems, which has been the tendency during recent years, seems to have passed and gradually more attention is being given to work of a fundamental character from which valuable results may be expected to develop. Some of the more prominent of these fundamental studies are summarised.

DURING the past few years there has been a tendency at the National Physical Laboratory to give priority to short term problems having a bearing on national productivity, but the urgent need for this policy appears to have passed, and but for the international situation we would now be hoping to devote our main energies to larger questions of more permanent interest. Fundamental work in all sciences has been forced into second place for a very long time, and it would be a thousand pities if we were at this time to be forced by a new armament programme to devote our time to the humdrum details of those engines of war of which we have every reason by now to be thoroughly tired. It is to be hoped that the necessity will not arise, and that we shall be free to perfect the more interesting and more valuable lines of work that are developing.

Mechanical Properties of High-Purity Iron

Prominent among these is the systematic examination of the mechanical properties of solid solutions, particularly those of iron. This has made a great step forward during the past two years, and 25 lb. melts of alloys containing not more than 0·01% of any unwanted impurity are now regularly made. The control of composition that has been achieved should make it possible to attack many obscure problems, and at the moment brittleness is engaging most of our attention. The magnitude of the chemical problem involved in this work is not generally realised. To estimate impurities of the order of 0·01% the methods of analysis must be reliable to within 0·001%. Very few of the ordinary methods meet this standard. At this level of impurity, interferences of one element with the determination of another are very common: each method has needed to be carefully scrutinised, and in several cases new procedures have had to be devised. This work in itself is a useful contribution to knowledge.

The choice of 0·01% as the maximum allowable amount of impurity was quite arbitrary: many of the important impurities, such as carbon, oxygen and nitrogen, are well below this limit, and generally below 0·003%. The iron as a whole has a purity of about 99·97%. Whether this standard of purity is sufficient remains to be found. There is evidence that some impurities exert significant effects on the properties of iron when the quantities are of the order of 0·001%, and one of the immediate tasks is to determine the nature and importance of these effects. When this has been done, the effects of the major alloying elements can be studied one by one, with the reasonable hope of securing data that are not confused by uncontrolled factors.

Iron of this degree of purity is subject to two types of brittleness, one an intercrystalline brittleness that is most readily developed when the grain is coarse, and the other a transcrystalline cleavage. They do not appear to be influenced in the same way by alteration of composition, but both types are favoured by lowering the temperature, and the study of the way in which the change from tough to brittle takes place as the temperature is lowered is expected to throw some light on the nature of both types of brittleness. At room temperature and under normal conditions of stressing pure iron behaves as a soft and very tough material but, as is well known, the temperature at which the change from a tough to a brittle behaviour occurs is strongly influenced by the stress system.

Titanium and Its Alloys

Interest in titanium has grown rapidly during the past two years, and the investigation of the constitution of its alloys is coming to be one of the most important of our researches. The metal dissolves oxygen very readily and its general properties are profoundly modified. Pure titanium free from oxygen is soft and ductile, but as little as 1% of oxygen changes it to a material which is as hard as a hardened medium carbon steel, but decidedly more brittle. If titanium develops as an industrial metal, it is likely that oxygen will play the same part in its metallography as carbon plays in the metallography of steel. It will then be most important that its behaviour be thoroughly understood. The examination of the effect of oxygen in titanium has been hampered by the difficulty of determining its quantity, but this problem has been solved by the successful application of the vacuum fusion method, substantially as applied to steel, but with modifications of detail designed to ensure that the whole of the oxide present in the metal is reduced. Considerable experience in the analysis of samples of titanium has now been gained, and confidence in the results is such that an investigation of the constitution of titanium-oxygen alloys has been set in hand. The extension of this work is planned to ternary systems containing carbon, nitrogen, and such other alloying elements as are likely to appear in commercial titanium. Most samples of titanium made by the magnesium reduction process contain 0·1 to 0·2% of oxygen: some of them contain much more: and investigation of the alloys of titanium is likely to be rather misleading unless the presence of this oxygen is realised and its effects are appreciated.

Plastic Deformation of Metals

Experimental work on the nature of plastic deformation is developing in an interesting way. The work began with an attempt to study creep in metals by X-ray methods, in the course of which it was found that slow

deformation, whilst being essentially a slip process, differed in its nature from fast deformation, and resulted in a different kind of disintegration of the metallic crystal. It has been continued by applying the electron microscope, the phase contrast microscope and the polarising microscope to a creeping specimen of aluminium. With the electron microscope it was found that the slip process is something more complex than a simple sliding of blocks of metal over each other; with the phase contrast microscope it was found that, besides the major slip bands observed in the ordinary metallographic microscope, there are innumerable finer movements involving displacements of not more than 100 angstroms: with the polarising microscope it was found that in some circumstances the crystal during creep will break up into a clearly marked array of slightly disoriented blocks that turn relatively to each other as deformation proceeds. The whole picture of plastic deformation is becoming more detailed and more involved. Many of the complexities are no doubt due to the mutual reactions of adjacent grains in an aggregate. Interesting results should follow from a comparison of the behaviour of single crystals and polycrystalline samples of the same material. The yet unsolved problem how to derive the properties of a polycrystalline aggregate from the fundamental properties of the single crystal, may perhaps be gradually approached by this means.

Other Work in Progress

The thermodynamics section that was set up two years ago has done useful work on the reducibility of oxides and chlorides of titanium and vanadium and is at present interested in the question of calculating deoxidation equilibria at high temperatures from the data provided by the methods of thermochemistry. The deoxidation of iron by manganese is being used as an example: in this system the oxides of manganese and iron are completely soluble in each other and it becomes possible to calculate the composition of the oxide that should be formed in given circumstances and to check the result by experiment. At the moment of writing a pleasing measure of agreement is being obtained. During the past year a powder metallurgy laboratory has been equipped and will be used to study the constitution of alloys of metals of high melting point. The constitutional work of the Laboratory has not been emphasised in this article, but it is nevertheless always going on, and is likely to increase somewhat in importance in the near future if circumstances permit a normal development of the work.

British Ceramic Research Association

A. E. Dodd, Ph.D., M.Sc., F.R.I.C.

Head of the Associations Research Department

Fuel economy has become a permanent feature of industry, but, in addition, there is a growing awareness of the potential savings of time which are rendered possible by the use of refractory materials of low conductivity and low thermal capacity.

AN important part of the Association's work on refractory materials has been concerned with research on heat insulating materials. For over ten years the Association has been engaged on a continuous programme of work on the development of insulating refractories and on improved methods for their testing. Of special interest, perhaps, and of wide general application, has been the study of the relationship between the heat transfer through a refractory and its texture.

It has been induced, from experimental data, that the thermal conductivity of a pure crystal is inversely proportional to the absolute temperature. To apply this conception to heterogeneous refractories requires cognisance to be taken of the fact that refractories, in general, consist of crystalline and glassy matter interspersed with air-filled pores.

It is also known that, as the temperature increases, the conductivity of a material in the vitreous state approaches that of the same material when crystallised. Since insulating refractories are used at relatively high temperatures this fact is of importance, for most refractories contain a considerable amount of glassy bond. However, the thermal conductivity of the actual solid material of fireclay insulating refractories is lower than that of other refractory materials, and L. R. Barrett[1] has pointed out that the thermal conductivity of the solid matrix of an insulating product is significant if the porosity is high, although the conductivity is quite closely proportional to the bulk density within the normal range of porosities.

1 *Trans. Brit. Ceram. Soc.*, **48**, 235, 1949.

The theoretical approach to heat transfer through porous bodies must treat such bodies either as a solid continuum with discrete air cells, or as solid grains suspended in air; the normal insulating refractory behaves in a fashion intermediate between these two extreme cases. There is an obvious difference in texture, however, between say, a diatomaceous insulating material and a fireclay insulating refractory, and the thermal conductivity of a diatomaceous material is normally lower than that of a porous fireclay product of the same porosity; the order of this difference is almost 1 B.T.U./sq. ft./hr./° F./in., and is of sufficient importance to merit a more detailed study of the factors on which the low conductivity of diatomite depends. Primarily, the reason must be associated with the microporous structure of diatomite, which consists of the siliceous skeletons of minute living cellular matter. Advantage may be taken of the outstanding insulating value of diatomite only at temperatures up to about 900° C. to 1,000° C., however; if used at higher temperatures the structure of the diatomite is lost and serious shrinkage takes place. For this reason, diatomite is used only as a backing material, normal refractories being used for that part of the furnace lining actually exposed to the hot combustion gases. The bulk density of diatomite bricks is generally of the order of 30 lb./cu. ft. and the crushing strength is low, usually about 250 to 300 lb./sq. in.

Fireclay insulating refractories can be used at higher temperatures, and small furnaces can be built entirely of this insulating material. By far the most common

method of producing these refractories is by the addition of a combustible material to the fireclay batch which is then extruded, dried, and carefully fired. In experimental work at the Research Association's laboratories, the dry-press process of manufacture has also been used; the process is of special value if prefired porous clay is used as grog. It has been found that many materials that can be made to produce very light porous bodies, cannot be used directly for the manufacture of bricks or large shapes owing to their inherently high shrinkage during drying and firing; such materials can be utilised, however, as a source of porous grog which can then be bonded with a small amount of refractory clay and dry-pressed to the desired shape. Experience has shown that insulating bricks made in this way are rather heavy, and consequently of less value from the standpoint of heat insulation; the increased bulk density is in part attributable to a decrease in the porosity of the grog as a result of crushing during the pressing operation. However, dry-pressing can have some advantages over other methods of manufacture in that the pressed bricks retain their shape during drying and firing more satisfactorily than do extruded bricks containing a high proportion of combustible.

In the Research Association's laboratories, insulating refractories have been made with a dense slag-resistant face. Such products are made by placing a layer of a suitable firebrick mixture in the brick mould, then pouring the porous mixture onto the loose surface of the facing layer; when this composite mass is pressed, a very strong union between the two layers results. It is necessary to adjust the porous and the dense mixtures so that they have approximately equal shrinkages when the composite brick is dried, and again when it is fired.

In the first laboratory trials with this production technique the dense facing was made of fireclay grog; it would clearly be more attractive however, to use a more specialised facing that would offer still greater resistance to slag attack and fusion, but it was found that materials such as calcined kyanite and silicon carbide had such small shrinkages that it was difficult to match them with the porous body of the brick. Subsequent research has proved, however, that it is possible to control the shrinkage of kyanite coatings by incorporating a predetermined amount of uncalcined kyanite in the mix.

The ability of any refractory material to withstand sudden changes of temperature is determined by the magnitude and uniformity of its thermal expansion, by high mechanical strength and by low elasticity. Insulating refractories have low strength, but low elasticity. The higher spalling resistance of some insulating products is probably associated with their low elasticity and their ability to develop a somewhat loose structure if the internal stresses become too great.

Mention may also be made of the development in the Research Association's laboratories of a simple test, originally suggested by the Gas Light and Coke Co. Ltd., for obtaining comparative data on heat insulating values. The standard apparatus for determining thermal conductivity is of the calorimeter type; it is elaborate, demands high manipulative skill and is time-consuming. The simple comparative test now evolved is based on the measurement of the current consumed by an electric heater placed inside a bored-out test-piece when the internal temperature is maintained at a constant value. The many factors that may influence the result of a simple test of this type have been investigated; the most important are inadequate sampling (which would apply equally to the standard test), departure from the prescribed bore, a change of heater size, and exceptionally low emissivity. A close study of the test has established that it is a quick, practical, alternative to the standard method, and well suited to use under industrial conditions.

Standardisation of Sizes of Tubes made from Special Refractories

THE Sintered Oxides Sub-Committee of the Inter-Service Metallurgical Research Council (Ministry of Supply and Admiralty) has held discussions with manufacturers with the aim of improving the supply of *tubes* of special refractory. At present, delay in the delivery of these items arises mainly from the large number of different sizes which are demanded by the users, making it difficult for the manufacturers to maintain stocks.

It has now been agreed to concentrate on production of a range of sizes which are to be regarded as standard henceforward. The sizes adopted have been chosen after careful consideration of the demand for tubes in recent years, and it is hoped that they will fulfil the requirements of most users. The manufacturers have undertaken to maintain stocks of the standard sizes, so that orders may be met from articles in stock.

The success of this attempt to provide better supplies of tubes will depend of course on the wholehearted co-operation of the users. In particular, those employing large numbers of tubes are asked to standardise their sizes at the earliest possible moment and those designing new apparatus are asked to use standard sizes wherever possible. Production of the standard items will commence immediately. Non standard tubes will be obtainable, but their delivery may still be subject to delay.

Tube standardisation details are as follows:—

1. The materials involved* are: (i) Impervious alumina and (ii) Impervious mullite for use at temperatures up to 1700 °C.

2. The cross-sections of the standard sizes† are:—

Internal Diameter mm.	5	10	15	25	40	50	65	75
Wall thickness mm.	1·5	2·0	2·5	3·0	4·0	4·5	5·0	5·5

All tolerances will be as stated by the individual manufacturers.

3. All standard tubes will be available in lengths up to 900m.m. with both ends open, and also with one end open and the other hemispherically closed.

Thermocouple insulator. Whilst dealing with tube standardisation, the opportunity has been taken to introduce a standard cross-section of impervious alumina twin-bore thermocouple insulator. It will have an oval section, with 3·5mm. major and 2·0mm. minor axes, and 1mm. bores, 1·5mm. between centres.

It must be emphasised that the full effect of standardisation will not be evident until the maximum possible number of users are able to adopt the new sizes and until the manufacturers are able to build up stocks. Neither of these processes can be brought about immediately, but it is hoped that the standardisation scheme will be working efficiently in the very near future.

* The descriptions of the two classes of special refractory are those agreed by the manufacturers, who will provide details of the properties of their products on request.

† Metric sizes are adopted in view of the present trend in that direction and because many special refractory tubes are used in research, where the metric system is usual.

The British Non-Ferrous Metals Research Association

Creep and Fatigue Testing Equipment in the Laboratories

By J. McKeown, D.Sc., M.I.Mech.E., F.I.M.*

(Communication from the British Non-Ferrous Metals Research Association)

The Research Associations' activities in the study of creep and fatigue have expanded considerably in recent years and the present paper describes creep and fatigue testing equipment evolved and the techniques suitable for testing a wide range of non-ferrous metals over a considerable range of temperatures.

THE determination of the stress-strain-time-temperature relationships of metals is in general covered by two main divisions of mechanical testing, namely, creep testing and fatigue testing. Each laboratory engaged in such determinations has developed its own techniques of testing and forms of testing equipment best suited to the types of materials it has to deal with, the choice being also to some extent dictated by the available capital and manpower.

In the Mechanical Testing Laboratories of the B.N.F.M.R.A. it has been necessary to evolve equipment and techniques suitable for testing a wide range of non-ferrous metals over a wide range of temperatures. In what follows a brief description is given of this equipment and some of the reasons which have directed its development are explained. In general, these Laboratories have not been engaged on fundamental research on the mechanism of creep or of fatigue but rather on the comparison of these properties over a range of compositions and temperatures in alloy systems. Some work is, however, in hand on a fundamental study of the effect of metallurgical structure on the creep and fatigue of lead and of aluminium. For the work on lead special equipment, described below, has been developed.

GENERAL CONSIDERATIONS

In creep testing of non-ferrous metals and alloys the temperature range to be covered is from room temperature to about 750° C. Continuous creep may be observed at reasonably low stresses at room temperature in such materials as lead and tin and their alloys, at 200° to 300° C. in aluminium base alloys, and in the range 300° to 600° C. in copper base alloys. In consequence the equipment developed in these laboratories for elevated temperature creep testing has been designed to cover the complete range up to 750° C.

In creep tests at elevated temperatures on wrought non-ferrous metals the stress seldom if ever exceeds 25 tons/in.^{2}. For materials in rod this has been provided for by machines capable of applying a load up to 2·5 tons. In wrought materials a specimen diameter of 0·357 in. (cross-sectional area 0·1 sq. in.) is amply large. For materials in the cast condition this diameter is still suitable provided the grain size is not too large. If the grain structure of the material to be tested is coarse a diameter of 0·564 in. is more suitable and the 2·5 ton machine can apply stresses up to 10 tons/in.^{2}, which in general are sufficiently high for such a material. Other machines capable of applying a load up to 1 ton are provided for tests on wrought materials in the form of sheet on strip.

In the Association's experience the useful limit of strain measurement is set by the well known lack of close reproducibility of creep characteristics in materials as normally produced. Such lack of reproducibility is due to many causes amongst which may be cited variations in composition, in grain size, in degree of working in the case of cold or hot worked materials, in working temperature in the case of hot worked materials, in rate of cooling in cast materials and, in general, in departures from true homogeneity. Even in specimens taken from the same bar of worked material or from the same melt in cast materials considerable differences may be obtained in the steady rate of creep and in the plastic creep strain at a given time, although the specimens may have identical properties when subjected to tensile and other short time tests. The differences may on occasion be sufficient to produce changes in creep rate of as much as 100% and in consequence differences in creep properties between two materials cannot be properly assessed on the results of a small number of tests but should be determined using a range of specimens containing all the probable variations likely to result from normal production methods. Even when materials are produced in the laboratory under much more strict conditions of control than are economically possible in commercial production, the degree of reproducibility is seldom good enough to permit conclusions to be reached from single tests. The long duration creep test is about the most sensitive test tool available to the metallurgist and engineer for indicating those small differences in materials not shown by the more common short time tests. It is our experience that the reproducibility of creep test results has seldom, if ever, been good enough to justify measurement of strain to better than about 1×10^{-5}.

The development of the creep testing facilities in these laboratories has, therefore, been governed by the need for large numbers of test results. At the present time there are 76 machines available for tests at elevated temperatures and a further 176 units are in use for room temperature tests. The equipment was designed, and most of it was made, in the Association's laboratories*.

* Head of Mechanical Testing Section, British Non-Ferrous Metals Research Association.

* The elevated temperature creep equipment designed in these laboratories and described in this paper is now manufactured by The Delancey Tool and Engineering Co., Delancey Street, London, N.W.1, to whom enquiries may be addressed.

The use of multiple machines and simple methods of temperature control and of strain measurement has provided extensive creep testing facilities at a very moderate capital cost.

Sensitivity of strain measurement in creep testing is intimately connected with the degree of specimen temperature control and also to some extent with the accuracy of temperature measurement. Temperature is controlled in the B.N.F. equipment to $\pm$ 1° C., the temperature at any time being read to 0·25° C. For a temperature change of 1° C. the thermal expansion of a specimen of say a bronze is about $1{\cdot}5 \times 10^{-5}$ in. per in., while with an aluminium alloy it may be as high as $2{\cdot}5 \times 10^{-5}$ in. per in. For the degree of temperature control used therefore it is sufficient to measure the strain to $\pm\ 2 \times 10^{-5}$.

In those cases where the value of the steady rate of creep is required, two methods of determination may be adopted, either by the use of a high sensitivity extensometer (coupled with very close temperature control) over a short time or by the use of a less sensitive one over a long time. Thus in Fig. 1, when the creep curve has been defined by a reasonable number of points, the steady rate of creep is $\frac{\epsilon_2 - \epsilon_1}{t_1}$ A reasonably reliable minimum value of strain measurement is 5 times the smallest count of the extensometer. Thus in the B.N.F. equipment using a 5-in. gauge length specimen the reliable minimum value of $\epsilon_2 - \epsilon_1$ is 1×10^{-4} strain. When the rate of creep is constant for at least half the test time it is therefore possible in this equipment to determine the rate of creep in a 1,000-hour test to 2×10^{-7} strain per hour.

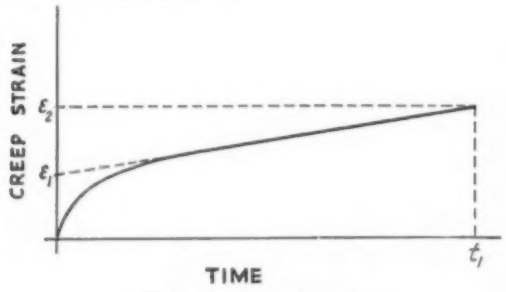


Fig. 1.—Creep Curve.

For design purposes the total plastic strain in a given time is required. For example, B.S.S. A23 calls for equipment capable of measuring total plastic strain down to 0·001 (0·1%) in periods up to 10,000 hours. In the B.N.F. equipment the least count of the extensometer is 2×10^{-5} strain, i.e., one-fiftieth of 0·001 strain.

Another important factor in creep testing which calls for consideration is the length of testing time used. The long-time creep behaviour of a material cannot be predicted with confidence from the results of short-time creep tests, largely because our knowledge of the factors determining creep behaviour of a given material is still very imperfect and partly because the materials tested may undergo structural changes during long periods at elevated temperatures, which changes may profoundly modify their creep behaviour. For example, a cold worked material may undergo slow recovery or even recrystallisation. If the latter process is really slow it will not be possible to detect its occurrence by examination of the creep curve. On the other hand precipitation may occur under the action of the temperature combined with the creep deformation. It is clear that when a material is to be put into service use in a condition which is unstable at the temperature involved, design data on its creep characteristics can only be determined satisfactorily by long duration tests at this temperature and at stresses of the same order as the service stresses.

Where an approximate classification of a range of materials is required short duration creep tests have some value but such tests may only be looked on as sorting tests and final assessment of the creep characteristics of the better materials must be based on long duration tests.

In the Association's laboratories sorting of materials is carried out by tests of 500 hours' duration, while design data on the promising materials is obtained from tests carried out for 5,000 to 10,000 hours using a range of stresses. During the War years design data was frequently required for materials which were to be incorporated in equipment the operating life of which was to be only 500 to 1,000 hours. For such materials 1,000-hour tests were satisfactory with only occasional tests carried on beyond this time.

The Research Associations Creep Testing Equipment

A.—*Elevated Temperature Equipment.*

This may be divided into three main types :—

(*a*) Individual units with long levers.

(*b*) Multiple units with short levers.

(*c*) Multiple units using direct loading.

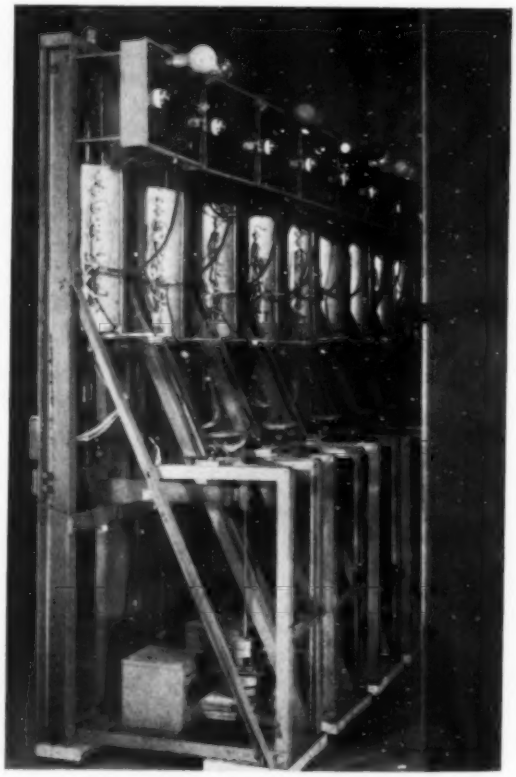


Fig. 2.—Long lever creep units.

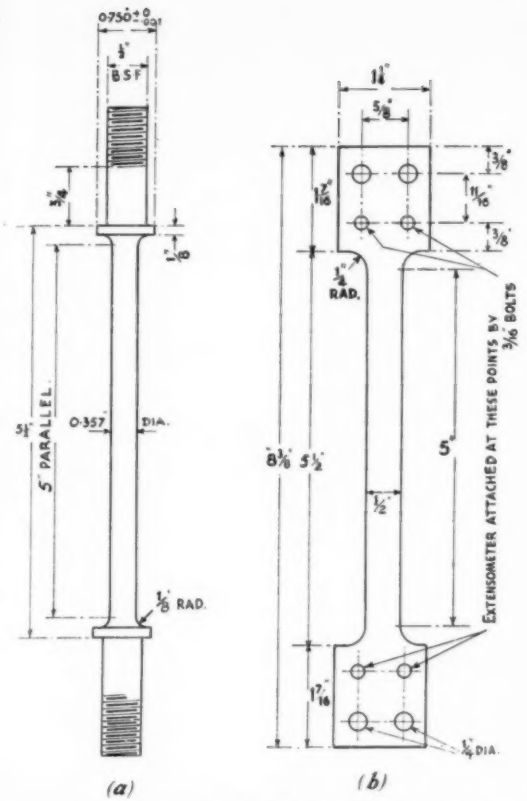


Fig. 3.—(a) Round, and (b) Flat creep test specimens.

(*a*) The long lever individual units, lever ratio 10 : 1, are capable of applying any load up to 2·5 tons to the specimen. A photograph of these machines is shown in Fig. 2. Twelve of these units are in use and in each the actual lever ratio has been determined experimentally. All fulcra are of hardened steel. The load applied by the lever and scale pan alone is 84 lb.

The loading shackles are made in two sections to break heat flow, the portions inside the furnace being of heat-resisting steel. The connection to each end of the specimen is through a nut in a spherical seating, to provide some measure of alignment and axial loading. Axial loading is also assisted by a double pin joint above the furnace and by a single pin joint at right angles to the knife edge below the furnace. The round specimen used in these machines is shown in Fig. 3A, the screwed ends engaging with the nuts in the spherical seating of the loading shackles. The extensometer, similar to that shown in Fig. 4, is attached to the collars and measures the separation of these collars as the specimen extends. The effective gauge length on which the measurement of creep is made is taken as the equivalent elastic gauge length which is dependent only on the geometry of the specimen and has been determined by using a steel specimen of high proportional limit. Extension measurements are made by advancing each micrometer spindle slowly by means of the ratchet until one click of the ratchet is heard. Using this technique the reproducibility of reading is ± 0·0001 in. and is independent of the observer, provided no undue inertia effects are introduced by moving the micrometers too quickly. The two micrometers enable extension readings to be taken on two sides of the specimen. Differences in these readings with increase in strain when loading the specimen indicate lack of axiality of loading and enable this to be rectified.

This extensometer is reasonably simple in construction and in attachment to the specimen and is, as near as possible in such instruments, trouble free. The range of extension that can be measured is not limited by the 1 in. range of the micrometers as, where necessary, distance pieces can be interposed between the micrometer spindle ends and the end of the measuring rods. In general, however, the range of the micrometers is ample as it corresponds to about 20% extension in the specimen. In the measurement of creep resistance at elevated temperatures it is, in most cases, only the first 1% or 2% of extension which is of interest from the view point of the potential user of the material.

The furnace consists of an inner tube of heat resisting metal, Inconel in most cases, with welded lugs at the two ends which carry the thermostat attachments. The tube is first covered with two thicknesses of asbestos paper and then coated with successive layers of alundum cement to a final thickness of about $\frac{1}{8}$ in. The windings are wound on the cement and covered by more layers of cement. The heat insulation consists of moulded semi-cylindrical sections of Newtempheit held in place by the outer casing of Birmabright. The windings are applied in three separate sections as shown in the furnace circuit diagram in Fig. 5. The top and bottom windings are in series and a variable shunt can be applied to either at will, thus enabling the ends of the specimens to be brought to the same temperature. While most of the heat goes into the specimens from the ends it is necessary, particularly at the higher temperatures to supply some heat by the centre winding, and this is done by supplying this winding at a suitable voltage from a tapped transformer. The temperature gradient in the specimen can easily be reduced to less than 1° C. For temperatures of 200° C. and lower current supply to the centre winding is found to be unnecessary. Also at low temperatures the main furnace supply voltage can be a lower value than the mains voltage, the appropriate value being obtained from the tapped transformer.

The thermostatic control operates the relay and mercury switch, placing the fixed resistance FR (Fig. 5) in and out of the circuit so that the windings operate between maximum and minimum current values. For

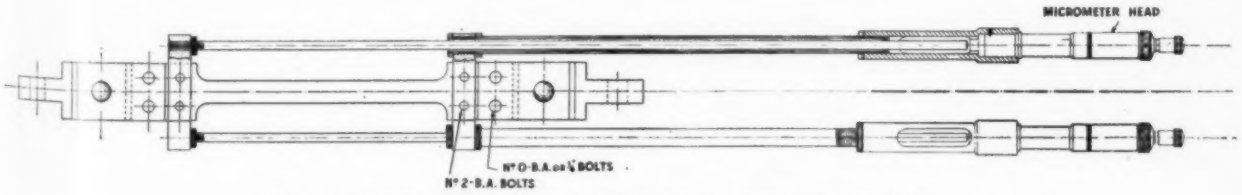
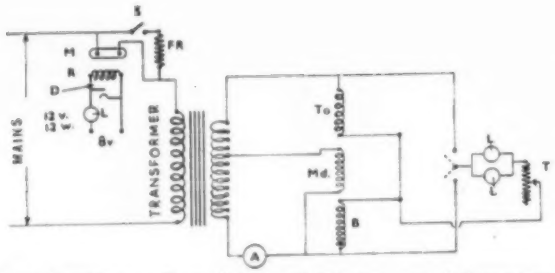


Fig. 4.—Extensometer shown with flat creep specimen attached.



A = Ammeter. D = dipper and mercury cup of thermostat.
M = Mercury switch. L = lamp.
R = Relay. T = fine trimming resistance.
F.R. = fixed Resistance.
To, B and Md = top, bottom and middle furnace windings respectively.

Fig. 5.—Furnace circuit for long lever creep units.

low temperature work the switch S is opened and the furnace current is operating "all on" and "all off."

The thermostat operates by using the differential expansion between the furnace tube and an Invar rod placed outside the furnace casing and hence substantially at room temperature. The differences in length resulting from changes in furnace tube temperature relative to the fixed length of the Invar rod are magnified by a 5 : 1 lever and the end of the lever pushes on a diaphragm on the underside of the high magnification element. The operation of this element will be clear from Fig. 6. An increase in length of the furnace tube results in contact being made between the mercury and the platinum wire dipper so short circuiting the relay and tripping the mercury switch. The circuit through this switch is broken and the fixed resistance FR (Fig. 5) is thus placed in the supply line. The resulting reduction of furnace current causes with time a decrease in length of the furnace tube and the reverse operation occurs. The voltage applied to the top and bottom windings of the furnace is set by the tapped transformer to make the complete thermostating cycle occur about once every minute. The specimen temperature is controlled over long periods to better than ± 1°C.

The temperature is measured by three chromel-alumel couples attached to the top, middle and bottom of the parallel reduced portion of the specimen. These couples are led to three four-point sockets fixed on the frame of the machine and a portable cold junction is plugged into each socket in turn, completing the circuit through the couple and the potentiometer. Thermocouple E.M.F. is read to 0·01 mv. which is equivalent to 0·25°C.

(*b*) *Multiple Units with Short Levers.*—These are mounted in three frames consisting of 16 testing units each, the frames being of a rigid angle-iron construction. The short levers have a nominal 5 : 1 ratio and each is separately calibrated for the determination of the exact value of the ratio. As in the long levers the fulcra are of hardened steel. The load applied to the specimen can have any value between 12 lb. and 1 ton. A photograph of one frame is shown in Fig. 7.

These units are generally used for tests on specimens machined from sheet or strip and the type of specimen used is shown in Fig. 3B. The extensometer is attached to this specimen by tight fitting bolts through the inner pairs of holes and the load applied through the outer pairs of holes. A close approach to axial loading is obtained by connecting the ends of the specimen to the loading rods by means of double pin joints with pins at right angles.

The furnace construction is very similar to that already described for the long lever units, except that the thermostat operates directly without the magnifying lever shown in Fig. 6. The furnace circuit diagram is shown in Fig. 8 and is seen to be a modification of the diagram for the long lever furnaces given in Fig. 5. The top and bottom windings are in parallel and trimming is carried out by a series resistance.

Thermocouple attachments are similar to those used in the long lever machines.

(*c*) *Multiple Units using Direct Loading.*—One frame of construction similar to that of the short lever units but using direct loading and consisting of 16 units is used for tests at loads insufficiently large to justify the use of levers. Furnaces and loading shackles are in other respects similar to those described in (*b*) above.

Creep Testing Procedure

When the specimen complete with extensometer and three thermocouples has been loaded into the furnace and connected to the loading bars a small load is applied

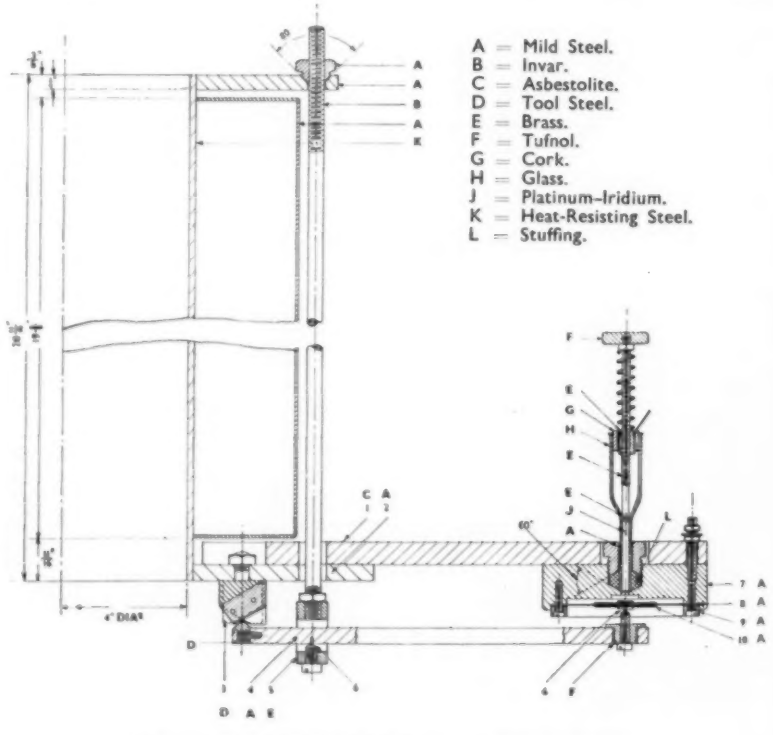


Fig. 6.—Thermostat for long lever creep units.

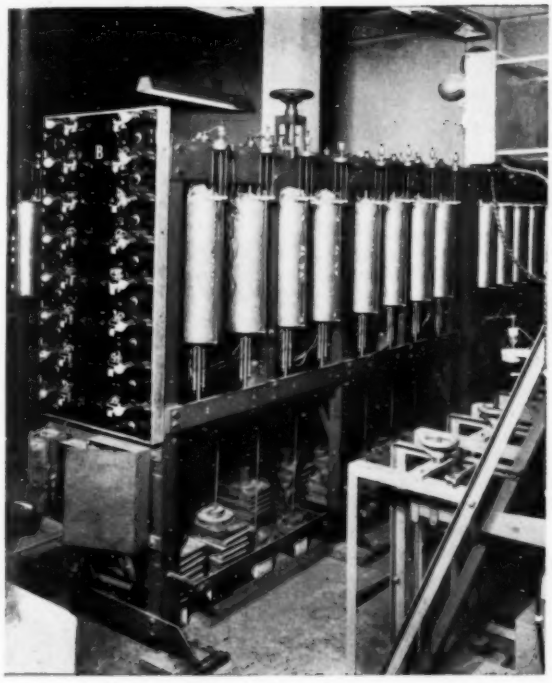


Fig. 7.—Group of short lever creep units.

and the furnace switched on. The temperature is brought up to the test value in about 6 hours, frequent checks being made on temperature gradient and any necessary adjustments made. When close to the test temperature considerable time must elapse before the effects of small adjustments are fully shown and in general the final adjustments are not made until the following morning, i.e., about 20 hours after first switching on. Load is generally applied after the specimen has been at the test temperature for about 20–24 hours.

Since none of the equipment described above employs the principle of a movable jockey weight on a lever it was necessary to develop an alternative method of increasing the load in steps at the commencement of a test. Such procedure is necessary to enable the Youngs Modulus of the specimen to be determined and its elastic deformation at the test stress to be calculated from this modulus.

The equipment used on all the units is shown in Fig. 9. It consists of a stand, which can be mounted on top of any creep unit, and which carried a proving ring with pulling up screw and hand-wheel. A connection is made between the bottom of the proving ring and the top loading bar of the creep unit. The load, whether at the end of a lever or directly under the specimen rests on a block of wood and is pulled up, through the specimen and the proving ring, by the screw and hand-wheel. The load is increased on the specimen in increments read on the dial micrometer of the proving ring. When each increment of load is applied readings are taken of the micrometers on the extensometer, thus measuring the extension of the specimen and so enabling the load-extension curve to be plotted. The slope of the elastic portion of this curve enables the Young's

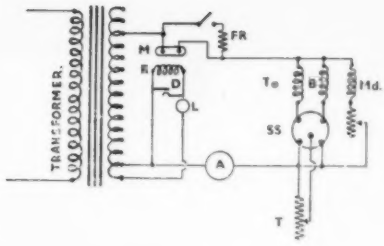


Fig. 8. Furnace circuit for short lever creep units.

A = Ammeter
M = Mercury switch
R = Relay
F.R. = Fixed resistance
D = Dipper and mercury cup of thermostat
L = Lamp
T = Fine trimming resistance
SS = Two roller action, double pole change-over switch
To, B and Md = Top, bottom and middle furnace windings, respectively.

Modulus to be calculated. When the full creep-test load has been applied to the specimen the nut at the top end of the top loading bar is screwed down on to the framework, and the loading equipment can now be transferred to another creep unit. The calibration of the proving ring can easily be checked from time to time in a tensile testing machine. The dimensions of the ring are such that 1,000 dial micrometer divisions equals 1,866 lbs. Provided the specimen has a limit of proportionality of 1 ton/in.^{2} or greater at the test temperature the result obtained for Young's Modulus by this apparatus is sufficiently accurate for test purposes. With higher limits of proportionality the accuracy is greater since the measured elastic extension is greater. Great accuracy in the determination of the Modulus is not really necessary as can be best shown by two examples.

Consider firstly a material with a limit of proportionality of, say, 2,000 lb./in.^{2} and a true value of the modulus of 12×10^6 lb./in.^{2} at the test temperature. The extension at the limit of proportionality of a specimen with an effective gauge length of 6 in. is 0·0010 in. This extension is only 5 times the smallest count of the extensometer ($\pm$ 0·0001 in.) and hence it is not possible to determine the Modulus to better than 2×10^6 lb./in.^{2}. Thus duplicate specimens may give 10×10^6 and 14×10^6 lb./in.^{2}. For such a material a suitable range of creep test stresses at the temperature would be 5,000 to 2,000 lb./in.^{2}.

At 5,000 lb./in.^{2} when $E = 10 \times 10^6$ lb./in.^{2} the elastic strain = 0·00050.

At 5,000 lb./in.^{2} when $E = 14 \times 10^6$ lb./in.^{2} the elastic strain = 0·00035.

At 5,000 lb./in.^{2} when $E = 12 \times 10^6$ lb./in.^{2} the elastic strain = 0·00042.

Thus the error involved in taking the

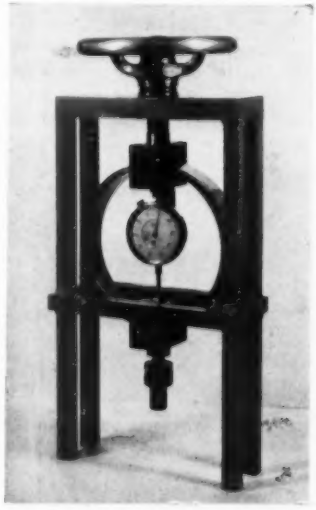


Fig. 9.—Loading equipment with proving ring.

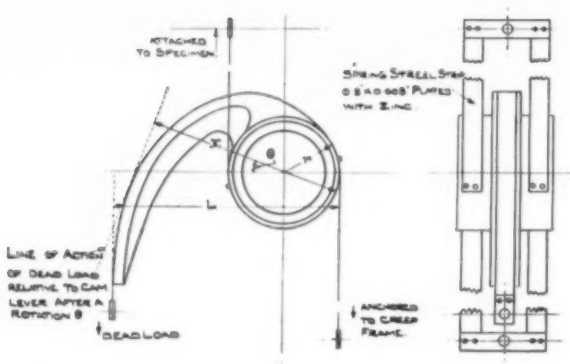

ATTACHED TO SPECIMEN
SPRING STEEL STRIP 0·8 × 0·005" PLATED WITH ZINC
ANCHORED TO CREEP FRAME
LINE OF ACTION OF DEAD LOAD RELATIVE TO CAM LEVER AFTER A ROTATION θ
DEAD LOAD

Fig. 10.—Cam lever for constant stress creep tests on lead.

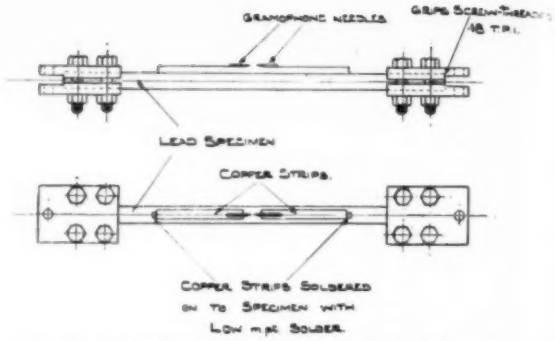

GRAMOPHONE NEEDLES
GRUB SCREW THREADS 48 T.P.I.
LEAD SPECIMEN
COPPER STRIPS
COPPER STRIPS SOLDERED ON TO SPECIMEN WITH LOW m.p. SOLDER.

Fig. 11.—Specimen for creep tests on lead rods.

mean value of E in each test instead of the actual measured value is 0·00008 strain. Although this is a substantial error in terms of the elastic extensions it is small in terms of the creep strains measured in the actual creep tests.

At a stress of 2,000 lb./in.^{2} the corresponding error is 0·00003 strain.

Now consider another material with a limit of proportionality of, say, 10,000 lb./in.^{2} and again a true Modulus of 12×10^6 lb./in.^{2} at the test temperature. The extension of the specimen at the limit of proportionality is now 0·005 in., i.e., 25 times the smallest count of the extensometer and in consequence the Modulus can be determined with much greater accuracy say between 11×10^6 and 13×10^6 lb./in.^{2}.

At a stress of 15,000 lb./in.^{2} the error in the calculated value of the elastic strain using 12×10^6 instead of 11×10^6 lb./in.^{2} for the value of the Modulus is 0·0001 strain in a total elastic strain of 0·00136. At 10,000 lb./in.^{2} the corresponding error is 0·00007 strain in a total of 0·00083. None of these errors is sufficiently large relative to the subsequent plastic creep strain to be of real importance.

In general, therefore, the apparatus described is sufficiently accurate for the determination of the elastic strain on loading, but if greater accuracy were ever required it could be obtained by measuring the Young's Modulus of typical specimens separately with standard tensile testing equipment for elevated temperature work.

As soon as the specimen has been fully loaded the extensometer readings are taken. Further readings are taken at 1, 2 and 4 hours and subsequently daily.

Stress Rupture Time Testing

The creep units described are also used for this form of test in which the specimen is subjected to a constant load and the time to produce fracture is determined. It is found convenient to use the same types of specimens for both creep and stress-rupture time tests as no modifications are required in the grips or loading shackles, as would be necessary were a shorter specimen employed. Each specimen is fitted with three thermocouples as before and the same degree of temperature control and uniformity is used. At the commencement of a test the full load is applied as a single increment.

The parallel length of the test specimen is marked off at 0·25 in. intervals and measurements made after fracture enable the elongation to be divided into its general and local components. General elongation is looked on as more important than local or total elongation as changes in it are indicative of changes in mode of deformation of the specimen with changes in stress and consequently of rate of creep.

When the specimen breaks the fall of the load trips a switch in the circuit of an electric clock. This clock makes one revolution in 168 hours and the time of failure of the specimens can be determined to the nearest half hour.

Room Temperature Creep Testing Equipment

This equipment is located in a constant temperature room, in which the air temperature is thermostatically controlled throughout the year at 20° ± 1° C. (68° F.). The humidity is not controlled. This room houses 8 frames each frame having accommodation for 22 specimens. The equipment is mainly used for testing lead and lead alloys and other metals of similar low melting point. As mentioned earlier continuous creep can occur in these materials at room temperature.

Much of the early work on lead and lead alloys was carried out on specimens of extruded pipe and cable sheathing but the more recent testing is being done on specimens of rod produced under carefully controlled conditions in a small extrusion press in the laboratory. This is being used for an extensive investigation of the effect of metallurgical structure and composition on creep behaviour.

In the early work on pipes and cable sheathing the testing was carried out under constant load conditions, i.e., as the specimen extended and its cross-section decreased the stress increased. In the newer work, on the other hand, the specimens are subjected to constant stress conditions of testing, these conditions being attained by the use of a constant stress lever. This lever, which has been described elsewhere,* is shown in Fig. 10. It is of simple construction enabling large numbers to be produced relatively cheaply. A total of 150 are in use. Referring to Fig. 10, as the specimen extends the lever rotates in space about the effective fulcrum which is the line of contact of the anchoring tape and the body of the lever, thus decreasing the lever ratio and hence decreasing the load applied to the specimen. While the principle is of general application it should be noted that the required contour of the cam is determined by the length of the specimen between the grips. In the case of the lead rods referred to above this length is 7 in.

* "A Simple Constant Stress Device for Creep Testing," by L. M. T. Hopkin, *Proc. Phys. Soc.*, 1950, 63B, 346.

The extension of the specimen is measured on a 5-in. gauge length. Light strips of copper, see Fig. 11, to which gramophone needles are attached are spot soldered to the specimen at points 5 in. apart and the relative movement of the points of the needles is measured with a cathetometer sensitive to ± 0·0002 in. (±0·004% strain on the specimen).

In creep tests on lead and lead alloys no attempt is made to measure the elastic portion of the extension. A generally accepted value for E for lead at room temperature is $2{\cdot}5 \times 10^6$ lb./in.^{2} and at a stress of 300 lb./in.^{2} the elastic extension would be 0·002% strain, i.e., less than the sensitivity of the measuring device. Consequently all creep curves are plotted as total strain against time. Since creep of lead and lead alloys is generally studied over an extension of 10% the inclusion of the small elastic component in the creep data does not introduce any significant error.

At the commencement of a creep test the load is applied without shock as a single increment, through a hydraulic jack.

In lead and lead alloys at room temperature, as in other materials at elevated temperatures, the changes in general elongation with changes in metallurgical condition and in stress or rate of creep are of considerable interest. Such changes are followed by marking the gauge length of the specimen, before testing, at 0·5 in. intervals.

FATIGUE TESTING

Elevated Temperature Fatigue Testing

This is carried out on machines designed and developed in the laboratories. The machine has been fully described in this Journal, Vol. 38, pp. 247-254, Sept., 1948, but the following brief description is given for the sake of completeness of the present paper. A photograph of the machine is shown in Fig. 12.

The specimen, which is loaded as a simple cantilever, is clamped at one end to the bed of the machine and does not rotate, so that measurement of its temperature becomes a simple matter. The outer end of the specimen carries a ball race, to the housing of which a small out-of-balance load can be attached. The housing is driven through a flexible coupling at a constant speed of 3,000 r.p.m. by a governor controlled D.C. motor. The out-of-balance weight offsets the centre of gravity of the rotating head so that the whole head becomes a rotating out of balance weight producing a rotating bending moment on the specimen.

If W_e = total effective out-of-balance weight
r = radius of rotation of this weight
and ω = angular velocity of rotating head.

then $F = \dfrac{W_e \, r \, \omega^2}{g}$ where F is the rotating load producing the rotating bending moment.

The magnitude of the rotating load can be changed by altering the magnitude of the small added out-of-balance weight. The determination of the actual magnitude of the rotating load is obtained from a calibration of the machine which is explained in detail in the original paper.

It is possible, and in fact desirable, to determine for each test specimen the relationship between the bending stress and the deflection of the specimen at the speed of testing used in the determination of the fatigue properties. This relationship, referred to as the dynamic calibration of the specimen, may, because of the absence of creep, be different from the statically determined relationship of bending stress and deflection.

Thus it may, particularly at elevated temperatures, consist of a relationship showing a range of proportionality of stress to strain, whereas under static loading conditions no range of proportionality may exist

The determination of the dynamic stress-strain-relationship for a material enables one to judge whether the maximum bending stress at the surface of the specimen is the stress, calculated on the assumption of the elastic behaviour of the material, or whether the stress in this region is relieved in each stress cycle by plastic deformation.

The machine has been used for testing at temperatures up to 750° C., which is the working limit of the present design of furnace, but this temperature might be raised by use of a suitable furnace and by provision of a spray form of lubrication for the ball race in the rotating head.

Room Temperature Fatigue Testing

In the past the greater amount of fatigue testing of lead and lead alloys has been carried out on direct stress machines, e.g., the Haigh machine. A suitable Wöhler type of test has been developed in the Association's laboratories.

In setting up a rotating cantilever specimen in a Wöhler machine it is seldom possible to arrange for the outer load carrying end of the specimen to run truly to better than ± 0·0002 in. With this setting the end of the specimen raises and lowers the applied load through a total distance of 0·0004 in. and in doing introduces considerable inertia forces when the speed of rotation is high. This inertia loading on the specimen is unidirectional and may easily be sufficiently high to give rise to unidirectional creep. The result of such creep is that the rotating deflection of the end of the specimen increases, which again introduces greater inertia forces and before long the machine trips, because of creep deformation of the specimen and not because of fatigue deformation. It was clear that the end load on the specimen should be applied in a way which would reduce the inertia effect to a minimum and this was achieved by using the form of spring loading shown in Figs. 13 and 14. The spring is calibrated in position on the machine and can be suited to the material, that is, to the range of fatigue stresses used. It will be seen

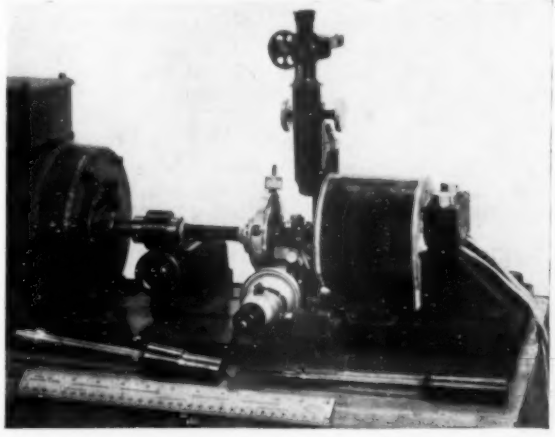


Fig. 12.—Elevated temperature fatigue testing machine.

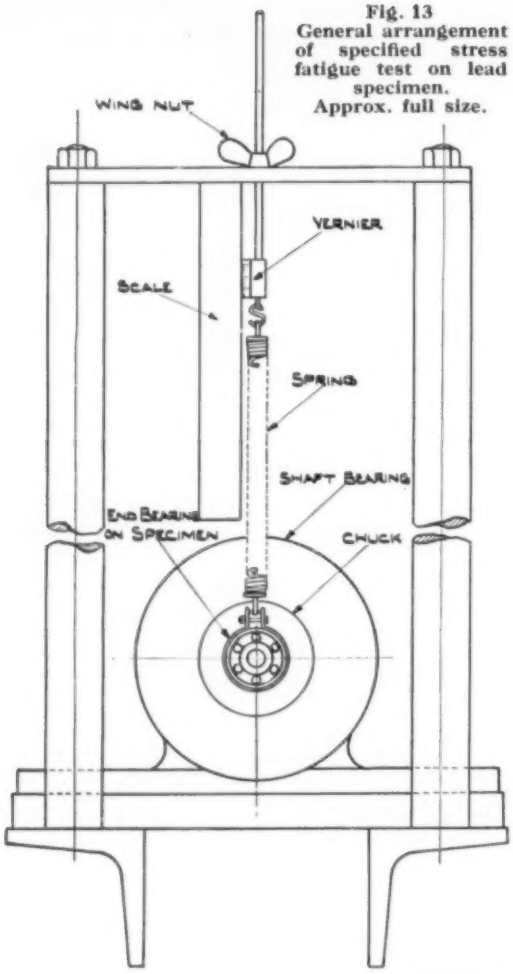


Fig. 13
General arrangement of specified stress fatigue test on lead specimen. Approx. full size.

that only the end coils of the spring are moving through the maximum out-of-truth deflection of the specimen, other coils having smaller movements, so that inertia loading is quite small. No inertia effect has been found

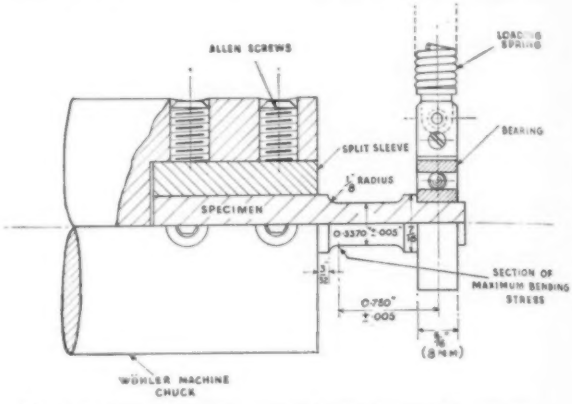


Fig. 14—Arrangement of specimen in specific stress fatigue test on lead.

in tests even on very high purity lead. The type of short specimen used, which is machined from extruded rod, is shown in detail in Fig. 14.

The type of test described above, which applies a constant bending moment, is suitable for work on specimens of either rod or pipe. The nominal bending stress can be calculated, assuming elastic conditions, and materials may be compared on the basis of this stress at the endurance limit. It is not possible to determine if this stress is a true elastic stress. Another form of test which applied a rotating deflection has also been used on specimens of pipe and was described in this Journal, Vol. 41, p. 135 and p. 219, January and February, 1950. In this test materials are compared on the basis of deflection of a standard test specimen at the endurance limit.

A contribution to the above series by Dr. D. F. Galloway, Director of the Production Engineering Research Association, has been unavoidably delayed and will be published in the next issue.

The Gas Council

Appointment of Research Committee

ONE of the major duties placed on the Gas Council, as the central advisory, co-ordinating and financial body of the gas industry, is the organisation and conduct of research.

The Gas Council announces that, to assist it in the discharge of this function, it has set up a Research Committee, to consist of three eminent men of science not engaged in the industry, three members of the Council, the President for the time being of the Institution of Gas Engineers, the Livesey Professor for the time being at Leeds University, and the directors of the Research Stations (probably three) which it is intended to establish.

The Chairman of the Committee will be the Chairman of the Gas Council, SIR EDGAR SYLVESTER, K.B.E. Two scientific members have been invited and have agreed to join the Committee, SIR ROBERT ROBINSON, O.M., F.R.S., and SIR CYRIL HINSHELWOOD, F.R.S.

The three members of the Gas Council who will serve on the Committee, and who all have scientific or technical qualifications, are MR. E. CROWTHER, Chairman of the Northern Gas Board. DR. R. S. EDWARDS, Chairman of the North Eastern Gas Board and MR. W. K. HUTCHISON, Chairman of the South Eastern Gas Board.

The President of the Institution of Gas Engineers for the current year is MR. F. M. BIRKS, C.B.E., Deputy Chairmen of the North Thames Gas Board, and the Livesey Professor at Leeds University is PROFESSOR A. L. ROBERTS, Ph.D., F.R.I.C.

The existing laboratories of the North Thames Gas Board have been recognised as the nucleus of the London Research Station and DR. H. HOLLINGS, F.R.I.C., who has for some years been Controller of those laboratories, has joined the Committee as Director-designate of the Station.

This Research Committee will be responsible for advising the Gas Council on all matters connected with research, for drawing up programmes of research and for reviewing progress from time to time.

The Iron and Steel Institute

Special Meeting in Glasgow

Nearly 400 members and ladies participated in what proved to be a most interesting and enjoyable four-days meeting in Glasgow, which, in addition to a technical session, included a Civic Reception, a Dinner and Dance, visits to many works, special sight-seeing tours for the ladies and an all-day excursion on the Clyde and a sail through the Kyles of Bute. This meeting will be a memorable one to all who attended, but here it is only possible to refer to it very briefly.

At the invitation of the West of Scotland Iron and Steel Institute, the Iron and Steel Institute held a Special Meeting in Glasgow on September 12–15 at which nearly 400 members and ladies attended. A very full programme had been arranged, which, in addition to a technical session, included visits to many works and sight-seeing excursions for the ladies, besides a Civic Reception, a Dinner and Dance, and a full-day sail along the Clyde and through the Kyles of Bute to Fairlie. The arrangements proved admirable and all who attended the meeting fully supported the President and Council of the Iron and Steel Institute in their expression of thanks to the Lord Provost and Magistrates of the City of Glasgow, the West of Scotland Iron and Steel Institute, and the Chairmen and members of the Reception, Executive and Ladies Committees; to the Scottish Iron and Steel Manufacturers and to the Directors of the Works who kindly invited Members and Ladies to visit them or had otherwise contributed to the arrangements made in Glasgow; to the authors of papers presented at the meeting and to all who contributed to making the meeting so enjoyable.

Members from eight countries, as well as ladies, were present at the official opening of the meeting, on the morning of September 12th, to receive a formal welcome by Mr. W. Barr, President of the West of Scotland Iron and Steel Institute in which he referred to the rather long time which had elapsed since the Iron and Steel Institute held its last meeting in Glasgow. He hoped the next meeting in Glasgow would not be so long delayed but expressed the sobering thought that it might be held under the flag of some board or other. He cordially welcomed members and the ladies and hoped that the programme, which had been prepared, would prove adequate and enable the meeting to be a memorable one. The meeting, which was held in the main hall of the Institution of Engineers and Shipbuilders in Scotland, then divided, the ladies to attend a Mannequin Parade and members a Joint Technical Session.

JOINT TECHNICAL SESSION

The Joint Technical Session was presided over by Mr. J.R. Menzies-Wilson, President of the Iron and Steel Institute, at which were presented and discussed the following papers: "The Reduction of Lump Ores,' by R. Wild and H. L. Saunders; "The Distribution of Temperature in Ingot Moulds and Its Relation to Ingot Structure," by I. M. Mackenzie and Andrèe Donald; "Ingot Heat Conservation; Time Studies from Casting to Rolling," by A. V. Brancker, J. Stringer and L. H. W. Savage; and "Ingot Heat Conservation: Mould and Ingot Surface Measurements," by A. V. Brancker. A further paper on "The Production of Steel Plates in Scotland," by J. A. Kilby, published in the September issue the Institute's *Journal* for this meeting, was not formally presented for discussion. Brief summaries of the papers are given below.

TIME STUDIES FROM CASTING TO ROLLING

By A. V. Brancker, Ph.D., J. Stringer, B.A., and L. H. W. Savage, M.Sc., F.R.I.C.

A RECENT survey of practice showed that fuel requirements of soaking pits at different works were 10–20 therms per ton of steel ingots over long periods, despite the fact that when tapped the steel has a total heat content about 5 therms per ton in excess of that required by the rolling mill. This is due, in some degree, to the impossibility, with present knowledge, of assessing what expenditure of fuel is incurred by the provision of a safety factor to ensure that, before stripping, the ingot will have a shell of sufficient strength to be handled without deformation. Without a knowledge of the temperature distribution at the time of charging the pit, it is also impossible to estimate the heat required and the time taken for the ingot to attain a sufficiently uniform temperature for rolling.

It is generally accepted, however, that there is a close correlation between track time and minimum heating time, where track time is taken as the interval between the end of teeming and the start of charging to the pits, and heating time as the interval between the end of charging to, and the starting of drawing from, the soaking pits. The appropriate correlation varies according to different authorities, e.g.:

Heating time = Track time

Heating time = Track time + 1 hour.

But it was noted from the survey referred to above that the average gross heating time varied from about 4½ to 14 hr. compared with track times of about 1½ hr. As in all cases there was a close correlation between fuel consumption and gross heating time, it appeared appropriate to investigate the flow of ingots from the steelmaking plant to the mills, and in this paper the authors describe observations made at three works where pit casting, bogie or car casting, and ground level casting were the respective practices. The information obtained from these observations and from a study of the routine records at one works has been analysed to find what improvements are possible, especially in reduction of time during which ingots can lose heat between teeming and charging to the soaking pit.

It is concluded that worthwhile savings are possible. In the case of one works it is estimated that feasible improvements, including systematic handling of ingots and a new system of keeping records, could result in an

increase in the soaking pit throughput of 18% made up of cold ingots. Alternatively, if extra ingots were not available, at least 18% of the pit capacity could be taken out of service whilst maintaining the present throughput.

MOULD AND INGOT SURFACE TEMPERATURE MEASUREMENTS

By A. V. Brancker, Ph.D.

ON the changes in heat content and temperature distribution of steel ingots between teeming and rolling depend the times required to solidify the steel sufficiently to permit safe handling, and to bring the stripped ingots to a uniform temperature in the soaking pits, but little is known of these changes. More or less arbitrary rules are adopted in the majority of works, stipulating a minimum solidification time, and relating the total track time to the subsequent heating time. Considerable variation in such times is found between different works, and this has an important bearing on the number of soaking pits required and on the fuel consumption per ton of ingots. It is desirable to obtain confirmation of actual measurements of the steel temperature at the various stages and, as these measurements cannot readily be made under normal casting-bay conditions, it was considered that attempts should be made, at first, to ascertain the surface temperature changes of mould and ingots, and to calculate the internal temperature distribution. If this proved practicable, it was thought that confirmation of the calculations might be possible by direct internal measurements on one or two ingots.

In this report of the work it is noted that surface temperatures have been measured at 36 positions on the four sides of a mould. These measurements extended from teeming to stripping. Two complete sets of temperature data were obtained for two different mould positions in a casting pit during two separate tap teeming operations. In each test, immediately the ingots were stripped, temperatures were measured with an optical pyrometer. Temperature continuity was therefore maintained until the ingots were charged to the soaking pits.

The first results obtained for the mould temperature measurements, using fixed thermocouples, showed considerable differences in the temperature, according to the position of the mould surface in the casting pit. The temperature of a long face adjacent to another mould was considerably greater than that adjacent to the pit wall. No attempt has been made to calculate the internal temperature distribution of the ingots, nor to assess the extent to which the non-uniform surface temperature may cause solidification to proceed more from one side than another. The technique of measuring the temperature of a freshly stripped ingot by means of a disappearing filament pyrometer was less satisfactory because of the rapid cooling of the ingot.

The author expresses the view that further work on mould temperature measurements is warranted to ascertain whether similar or greater differences will occur in other pit positions, and whether reproducible figures in any one position will be obtained with different casts.

THE REDUCTION OF LUMP ORES

By R. Wild, Ph.D. and H. L. Saunders, Ph.D., F.R.I.C.

THE output of modern blast furnaces has been augmented by the increasing attention paid to ore preparation. It has been recognised that an important aid to the efficient operation of furnaces is to fix a maximum size for the lumps of ore being charged. Gradually, however, it is being appreciated that screening out the fines, and only charging into the furnace ore which has been narrowly graded in regard to size, results in more efficient operation. As the authors of this paper state, if the ore charged contains a high proportion of fines, channelling and excessively high dust losses limit the blowing rate; on the other hand large masses of uncrushed stone will proceed down the stack with insufficient preparation within the lump.

Although it is not yet fully realised, the benefits to be obtained from proper sizing, in output and reduced coke consumption, are very definite. It is pointed out, however, that the physical and chemical properties of ores vary greatly, thus, there can be no one optimum size applicable to all ores, and an effort should be made to size each ore so that the lumps reach the hearth in an equally prepared state. The problem of correct sizing of ores is thus two-fold, firstly, to obtain lumps of such a size that the heating rate will neither produce disintegration, with subsequent dust losses, nor give inadequate heating at the centre; and secondly, to obtain the correct lump size so that gas penetration into the lump will be sufficient to effect adequate chemical reduction during the descent of the stack to prevent the hearth being chilled by endothermic reducing reactions. These two conditions may lead to conflicting values and it may be necessary to balance the one against the other, or the effect of one may be over-riding, thus making it possible to neglect the other.

The importance of adequate heat flow and gas penetration into the lump, and thus the necessity for the correct sizing of the ore, has been emphasised by many workers and in this paper the authors present the results of their investigation on the overall rate of heat transfer to the centre of ore lumps and the penetration of gaseous reduction. The factors studied are considered under the following headings: Nature of materials, moisture content, shape, size and degree of reduction. The ores used were Sorpresa Rubio, Zaccar, Grangesberg, Oxfordshire, Northamptonshire, Frodingham, Eaton and Sproxton, and, in addition, limestone.

Ore (i)	Porosity, % (ii)	Depth of Penetration of Reducing Gases, in. (iii)	Comparative Size in. (3-in Ouenza as standard) (iv)
Iron-rich hematites of high permeability			
(1) Ouenza	38·8	0·55	3
(2) Sierra Leone	43·0	0·8	4¼
(3) Djerissa	40·9	0·68	3½*
(4) Kelouchа	30·0	0·62	3*
(5) Konakry	28·8	0·8	4¼
Iron-rich hematites of low permeability			
(6) May-sur-Orne	18·2	0·4	2¼
(7) Cumberland	17·5	0·17	1
(8) Oued Zem	14·7	0·22	1¼
(9) Rif	12·5	0·15	¾
(10) Wabana	6·7	<0·1	< ½
Magnetites			
(11) Grangesberg	0·8	0·2	1
(12) Gellivare	0·8	0·13	¾
(13) Kiruna	0·4	<0·1	< ½
Permeable home ores			
(14) Oxfordshire	52·0	0·9	5
(15) Frodingham Limy	43·5	—	—
(16) Frodingham Silicious	45·5	0·87	4¾
(17) Northamptonshire Green	42·2	0·65	3¾
(18) Cleveland	25·9	0·6	3¼

* To obtain the comparative size for column (iv) the penetration depths for Djerissa and Kelouchа were compared with that for 2 in. and 1¼ in. Ouenza, respectively.

As a result of this investigation the authors conclude that :

The overall rate of heat transfer to the centre of the lumps does not vary very greatly with different ores and is not likely to be a critical factor except, possibly, in certain permeable home ores where the rate of heating inside the lump is retarded by endothermic reactions. In these ores calcination would have a beneficial effect.

In general, this work has established the fact that gas penetration into lump ores under blast-furnace conditions, and hence stack reduction, is limited by the porosity of the ore, and this factor should therefore determine the optimum size. The values of the limit of penetration are shown in the previous table, where the porosities are also given. The values in column (iv) are determined by a comparison of the penetration values for each ore with that of Ouenza and the adoption of a 3-in. lump of this ore as a standard. Certain deviations from this principle have been noted and reasons for them have been discussed. The actual sizes suggested for blast-furnace practice are comparative only, being based on a lump size of 3 in. for Ouenza. The extent to which these optimum sizes could be achieved in practice is linked with crushing efficiency and the ability to handle the fines produced.

With certain ores, wide variations in chemical and physical properties occur and, although an effort was made to secure a representative sample for investigation, the results for such ores should be interpreted in conjunction with analysis and porosity figures for the actual consignment to be used. The samples of magnetite ores employed were chosen to cover a range of ore types and thus to assess the effect of various gangue constituents.

THE DISTRIBUTION OF TEMPERATURE IN INGOT MOULDS AND ITS RELATION TO INGOT STRUCTURE

By I. M. Mackenzie and Andrée Donald

NUMEROUS investigations have been carried out to ascertain the cause of surface defects on ingots, such as cracks, double skin, blowholes, etc. As skin defects are produced by conditions prevailing during and immediately after turning, the solution of these problems awaits a more complete explanation of the effects of superheat and turbulence on the structure of the ingot, and the determination of the rate of abstraction of heat from the steel, and of the origin of stresses in the newly formed skin.

As part of an investigation into the mechanism of ingot cracking, experimental work on some of the controversial problems has been carried out and is reported in this paper. The principal questions for which answers were sought included :

1. What is the rate of transfer of heat from the ingot to the mould, and how does it vary with time.
2. Is the separation of the ingot from the mould continuous or discontinuous, and what is the extent of separation when the outer skin of the ingot is solidifying.
3. What is the structure of the ingot immediately below the surface and why does the mode of crystallisation change from the skin zone to the columnar zone.

The condition during the early stages of solidification was investigated by examining the structure of experimental ingots and by following the change in temperature distribution within the mould. A 3-ton ingot of mild steel, fully killed with silicon and aluminium, was sectioned and the sub-surface structure examined by sulphur printing and macro-etching. The effect of convection currents in the super-heated metal giving rise to banding of dendrites near the surface is discussed. An experiment to show the effect of " overkilling " with aluminium and silicon, in producing axial porosity, is described. The results obtained from experiments to determine the changing temperature distribution, within the ingot mould wall, are used in a calculation of the rates of heat transfer to the mould. It is shown that the rate of heat transfer is initially at a maximum value which may exceed 2,000 cal./sq. cm./min., the rate decreasing to about one-tenth of this value within 10 minutes. A graph method of successive approximations is used to calculate the rate of solidification of the ingot. It is shown that while the thickness of the solid skin may be given approximately by an expression of the form : skin thickness = constant $\sqrt{\text{time}}$, this function is only empirical and it does not hold over the first few minutes after teeming.

The factors controlling the rate of heat transfer to the mould are discussed with particular reference to the early separation of the ingot skin from the mould wall. The experimental data and the results of the approximate calculations are made the basis of hypotheses of the mechanism of solidification and of the formation of ingot cracks. The authors suggest that cracks are more like by to form during the first few minutes after teeming because of the rapid expansion of the mould and the consequent stretching of the newly formed ingot. A postulated mechanism is shown to explain the observed effects of superheat, teeming speed, and sulphur content on the incidence of cracking.

Continuous Casting of Steel Slabs

During the morning of September 13th, a film on the continuous casting of steel slabs by the Rossi-Junghaus process, at the Allegheny-Ludlum Steel Corporation, Watervlist, U.S.A., was shown at the Institution of Engineers and Shipbuilders in Scotland. The film was in colour and silent, and lasted about ten minutes. A short introductory explanation by Mr. J. Savage, of the British Iron and Steel Research Association, preceded the showing of the film and added greatly to the interest.

The continuous casting of steel into suitable forms for rolling has been studied in Britain for some years, but nothing so ambitious as that illustrated by the film has been attempted. The experimental plant depicted in operation was so designed as to provide a reciprocating movement to the mould to assist the delivery of the slab. A copper mould was used and arrangements made to maintain a water cooling service. The mould shown was designed for casting a steel slab 14 in. by 3 in.

The process is being applied to the casting of alloy steel slabs and, apart from the quality of the slab, which is claimed to be of a high order, the economic advantages are considerable, since very much less cropping is needed. Many questions were asked regarding the operation of the process which Mr. Savage clarified.

Special Functions

The enjoyment of the meeting was greatly enhanced by several functions that had been arranged, and which now must be a happy memory to all who participated. Notable of these was the Civic Reception given to Members and Ladies by the Rt. Hon. the Lord Provost,

Mr.V. D. Warren, M.B.E., T.D., and Magistrates of the City of Glasgow in the City Chambers, on the evening of September 12th. This function, and particularly the dance and concert which followed, was very much appreciated. Earlier the same day the Scottish Iron and Steel Manufacturers, with Mr. Alex. Williamson in the Chair, entertained Members and Ladies to luncheon in the Central Hotel, Glasgow. The charming welcome extended on behalf of the Manufacturers was responded to by Mr. Richard Mather, a Vice-President of the Iron and Steel Institute, on behalf of the Members and Ladies.

Probably the most outstanding success was the Dinner and Dance given by the Reception Committee in the Central Hotel on the evening of September 13th, with Sir Andrew McCance in the Chair. The toast of the City of Glasgow was proposed by Capt. H. Leighton Davies, C.B.E., a Vice-President of the Iron and Steel Institute, in which he referred to the industrial activities of the Clyde district of which Glasgow is the centre. The Lord Provost, Mr. Victor Warren, responded in an admirable speech in which he showed appreciation of the delicate political position of the iron and steel industry. The toast of the Iron and Steel Institute was proposed by the Chairman, Sir Andrew McCance, who referred to the changes which had taken place in the industry since 1927, when the Institute last met in Glasgow. In Scotland the industry had become more compact due to changes in national conditions. There were now fewer works, but production had substantially increased, much more pig iron was now produced from about one-quarter of the furnaces. Continuing, Sir Andrew said that in the public controversy that had raged around the iron and steel industry in recent years, he could not say that all speakers, in what they said, had used that simple criterion, truth, as a standard of reference. In any living and active business, he said, the process of improvement was a continuing one : how long it might continue was another matter. The response to this toast was made by the President of the Institute, Mr. J. R. Menzies-Wilson, O.B.E.

On September 14th, Members and Ladies were entertained to luncheon, at the Grosvenor Restaurant, by Colvilles, Ltd., with Sir John Craig, C.B.E., D.L., in the Chair. Sir John referred to the changes that had been effected in the iron and steel industry on Clydeside and spoke of the district as a nursery from which iron and steel works in various parts of the world implemented their staffs. In acknowledging appreciation to Colvilles Ltd., and for the pleasure of having Sir John Craig in the Chair, Mr. James Mitchell, C.B.E., referred to an early incident in his career when he offered his services to Colvilles Ltd. and to which he received the "encouraging" reply that his application was being considered. Since that time he had been rather worried with the thought that his application had come up for consideration at successive Board meetings, without reaching a decision, taking up valuable time ; he therefore took the opportunity to advise the Board, through Sir John, that he now withdrew his offer so that the firm would have no further trouble with it.

Works Visits

From a technical point of view, visits to works are frequently as useful to members of a technical organisation as the presentation of papers ; this is especially true of the works visits arranged in connection with this meeting. Properly organised works visits cannot fail to be profitable, and, since considerable care had been taken in the arrangements, the members found the visits to various Clydeside works, both interesting and informative.

Visits to works enable many aspects of production to be examined at first hand and comments made on the spot enable discerning visitors to draw useful conclusions. Thus, for instance, those concerned with the manufacture and use of iron and steel found the visits to many Clydeside iron and steel works of more than ordinary interest, as, for example, the seamless tube plant at the Clydesdale Works of Stewarts and Lloyds, Ltd., at Mossend, where that unique machine, the Pilger mill, attracted much attention. The ingot capacity at these works is 150,000 tons per annum, half of which is converted into tube at Mossend and the remainder transferred to other tube plants of the Company. The tube plants at these works is designed to produce plain-end oil-line tube of $10\frac{3}{4}$ in. to 16 in. dia., in rolled lengths up to about 80 ft. and thicknesses of $\frac{1}{4}$ in. and upwards.

The manufacture of high-grade alloy steels is the special function of Clyde Alloy Steel Co. Ltd., at Motherwell, and at these works members were able to see the two broad divisions of this Companies activities—steel castings and wrought steel. In the castings section all steel is melted electrically, being supplied from electric-arc or high frequency induction furnaces. A casting which attracted some attention was a stainless steel propeller about 12 ft. dia. Production in the wrought steel section is from three rolling mills and a forge.

A considerable number of members visited the Parkhead Works of Wm. Beardmore and Co. Ltd., which now covers over 95 acres, having grown from a small forge set up by Raith prior to 1830. Members were able to see the extensive modernisation and development carried out since the war, which has resulted in the entire reconstruction and enlargement of the steel foundry, the modernisation of the forging presses, the installation of many new machines and an increase in the scope of the works by undertaking the production of heavy engineering plant. Notable of the new developments being the production of forged-steel work-rolls for wide strip mills, sleeved back-up rolls and cast steel back-up rolls. There are facilities in the melting shop for making ingots up to 165 tons weight. In the forge it was fortunate for the visitors that the largest of the three forging presses, one of 6000 tons capacity, was in action. In the steel foundry, which is equipped to deal with castings up to 120 tons, much interest was centred on the moulding of a stern frame and on turbine casting casings. The armour plate department is equipped with a mill for rolling plates up to 14 ft. 6 in. wide, and, since the rolling of armour plate is an unusual sight, even to visitors directly concerned with the iron and steel industry, this mill being in action provided an added interest.

The plant at Lanarkshire Steel Co. Ltd., Motherwell, consists of open hearth furnaces, blooming and slabbing mills, heavy, medium and light section mills, and a fabricating department. The ingot output last year was 307,000 tons. The products of this plant cover a wide range and serve the requirements of industries engaged in shipbuilding, general engineering, steel construction ; the fabricating department is equipped for production

of all classes of structural work, including solid columns with slab bases. Colliery arches and props are a special line of manufacture by this Company.

The present works of Babcock & Wilcox Ltd., at Renfrew, was commenced in 1896 on the site of a small existing forge, to-day they occupy an area of more than 100 acres and employ a labour force of more than 6000. These works have nine main sub-divisions : the drum shop ; the plating, structural and crane shops ; assembly shop ; machine shop ; forge ; header factory ; the pipe shop ; the section factory ; the foundry ; and the research department. For such large and important works of this type little time could be given to any one sub division, but it is noteworthy that in the drum shop, steel plates up to 5 in. thick are bent in an 8000 ton press to form fusion welded boiler drums and other pressure vessels, the finished drums in some cases being over 40 ft. in length and weighing 70–80 tons. Each inch of the welded seams of these vessels is x-rayed ; for this work a special shop has been built containing a concrete chamber housing 2.000,000 volt X-ray equipment.

The plant of R. B. Tennent Ltd. consists of one acid open-hearth furnace of 50 tons rated capacity, four air furnaces with a combined capacity of 100 tons, two cupolas, one of 3 tons and one of 5 tons per hour capacity, and six oil and coal-fired annealing furnaces, each of 100 tons capacity. Included are also well-equipped machine shops with modern small, medium and large machine tools, Craven 36 in. roll turning lathes, wobbler milling machines and grinders. The firm specialises in the manufacture of iron and steel rolls for all ferrous and non-ferrous rolling mill applications, and roll castings up to 55 tons in weight. The firm are also manufacturers of "Adamite" alloy rolls. In 1946 the firm joined with several roll making firms to form the British Rollmakers Corporation, Ltd., in 1947, full technical collaboration was established with the Mackintosh-Hamphill Co., and the Ohio Steel Foundry Company in America.

The party visiting Tennent's also visited the works of Lamberton & Co. Ltd. and saw in various stages of construction a 32 in. by 20 in. by 60 in. three-high mill of unconventional design for sheet-rolling ; a 20 in. by 49 in. by 80 in. four-high mill for sheets ; heavy roller straightening machines to take joints up to 24 in. depth ; 400 ton load cold billet shears ; several horizontal forging machines up to 750 tons capacity ; and two heavy maxima presses of 1000 and 2500 tons load capacity.

At the Clyde Iron Works of Colvilles Ltd. at Tollcross, members were able to see a completely self-contained unit for the production of pig iron from raw materials, comprising coking plant, ore preparation plant, blast furnaces, steam raising and power generating plant, together with all other necessary services. They were entirely rebuilt in 1938–39 for the production of iron from two blast furnaces, but a larger furnace put into service in 1948 has involved the installation of additional plant and several items are still necessary to make full use of the furnace capacity. The surplus blast furnace and coke oven gases produced at these works are piped to the adjacent Clydebridge Steelworks, which now covers an area of some 140 acres and employs approximately 1,900 people. The steelworks consist essentially of two melting shops, a slabbing mill and a three-high plate mill, together with the necessary equipment of electric power stations, engineering shops and other auxiliary plant. In addition there is a fabricating shop well equipped for the production of welded structures.

Among the firms visited having a long history was that of Duncan Stewart & Co. Ltd., which, in the early days was mainly concerned with machinery for the textile industry, later machinery for the production and refining of cane sugar was undertaken and the scope subsequently increased to include plant for the production of beet sugar. The sugar machinery now manufactured includes cane milling plants, engines, diffusers, evaporators, vacuum pans and auxiliaries. In 1939, the company was acquired by Davy and United Engineering Co. Ltd. and now has available wide resources for the supply of heavy machinery, including the largest types of rolling mills, hydraulic presses, converters, metal mixers and ancillary equipment for the ferrous and non-ferrous metal industries. Several plants of these types were in course of construction at the time of the visit and much interest was centred on the models of two complete mills which are on order, one of which is to be shipped overseas when completed.

The River Clyde and the Kyles of Bute

One of the outstanding events of the meeting was the full day boat trip along the Clyde and through the Kyles of Bute, particularly as the last day chosen proved to be fine and sunny. Members and Ladies embarked at the Broomislaw on Queen Mary II which commenced her journey towards the Firth of Clyde. For several miles the activities on the river and in the many shipyards and docks attracted most attention and soon the steamer made harbour at the shipyard of John Brown & Co. Ltd., where both members and ladies made a hurried tour of these famous works. Re-embarking to resume the journey, a pointing figure in grey stone, which was quickly seen, commemorates Henry Bell who was the first in Europe to apply steam-power for marine purposes. As the steamer proceeded past Bowling, where the Clyde Canal finishes, the massive rock of Dumbarton came into view and soon Port Glasgow, with its high tenements and shipyards, appeared, followed by Greenock and a few minutes later Gourock, a reminder that the Firth had been reached and with it the stepping off place of the Western Isles. It was here that some of the finest lock and mountain scenery in Scotland unfolded itself. Sailing along the Gowal shore passing Innellan and making for the Toward Lighthouse the ruins of Castle Toward came into view, while on the other side of the Firth could be seen the red buildings of Wemyss Bay and Skelmorlie and the beginning of the "Measured Mile." Turning Toward Point the ship entered Loch Striven where the Island of Bute was shown in all its beauty and Rothesay offering a welcome. Gradually the steamer made for the Kyles of Bute with a view of what must be the most beautiful scenery of the whole Firth. Entering the Kyles, sailing between the small Burnt Islands and rounding the north end of Bute, Tighnabruiach could be seen on the right hand side. In a few minutes Kames Pier was passed and the ship steamed towards Ardlamont when the Island of Arran with its "Sleeping Giant" came into view. The steamer then headed for the open Firth, and, passing the Cumbraes, it turned towards the little town of Fairlie, where the jolly party disembarked and returned to Glasgow by train. Before reaching Fairlie, however, a very fitting tribute was paid to the organisers by Dr. Desch, and, with the singing of Auld Lang Syne, this memorable meeting came to an end.

Recuperative Furnaces

Use of Silicon Carbide Tubes Lends New Interest to System

THE use of silicon carbide recuperator tubes has undoubtedly done much to develop the recuperator principle in furnace work, and in the United States of late years large numbers of furnaces, especially in the heavy basic industries, have been built with tubes of Carbofrax bonded silicon carbide with very satisfactory results. This material has a thermal conductivity which is approximately ten times that of fireclay refractories; it does not fuse or soften, but dissociates chemically at a temperature above 2200° C.; it is less permeable to gases than fireclay or most other refractories, while at the same time its close-grained structure is highly resistant to flame abrasion.

As regards strength, this bonded silicon carbide has an average modulus of rupture of approximately 900 lb. per sq. in. at 1300° C., which is many times that of the best fireclay refractories.

Tubes in Unit Construction

The standard American Carbofrax tube is 5¾ in. external diameter, 4·1in. internal diameter, and 44 in. long; the English pattern differs slightly in the diameter, being 6in. external × 4¼ in. internal diameter. The general practice is for these tubes to be mounted between and supported by terminal walls which are built up of fireclay blocks to form a unit, as shown in Fig. 1, air being drawn or forced through the tubes while the products of combustion surround them. The spacing of the tubes is at 12¼ in. centres horizontally and 7 in. vertically, and the distance between the terminal walls may be from 3 ft. 8 in down to 2 ft. 10 in., according to the type of terminal block used for the walls, and whether the tube is only partially or completely inserted in the block. The shorter dimension is mainly used in installations where the gas temperature exceeds 1300° C., as it affords additional support for the tube. The wall blocks have to hold the assembly sufficiently tight to prevent leakage, and at the same time permit a certain movement to the tubes, these objects being achieved by grinding all the contacting surfaces of the blocks, and having them recessed or undercut to permit of sealing the block joints, as well as the tube ends with a cement which is resilient under working conditions.

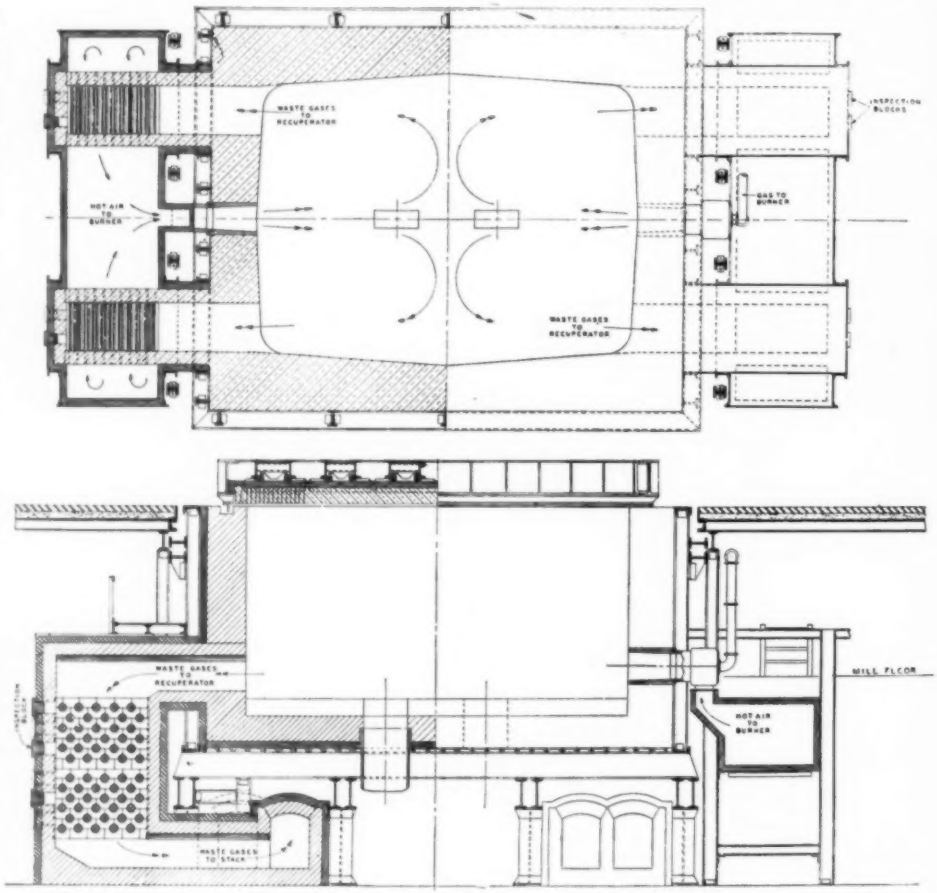


Fig. 1. Arrangement of four 2-pass recuperators for single-hole soaking pit.

Corebusters

In a plain recuperator tube such as was frequently used originally, the internal surface of the wall tends to be surrounded by a fairly thick film of hot air which does not allow the bulk of the air in the centre of the tube to come into contact with the hot wall. This is overcome by introducing into each tube a corebuster of suitable design, which considerably improves the efficiency over

Carbofrax is a registered trademark of the Carborundum Company.

a plain tube by increasing the internal convection area and promoting higher air velocities, thus increasing the rate of heat transfer. The corebuster that has been found most efficient is an "X" shape in lengths of

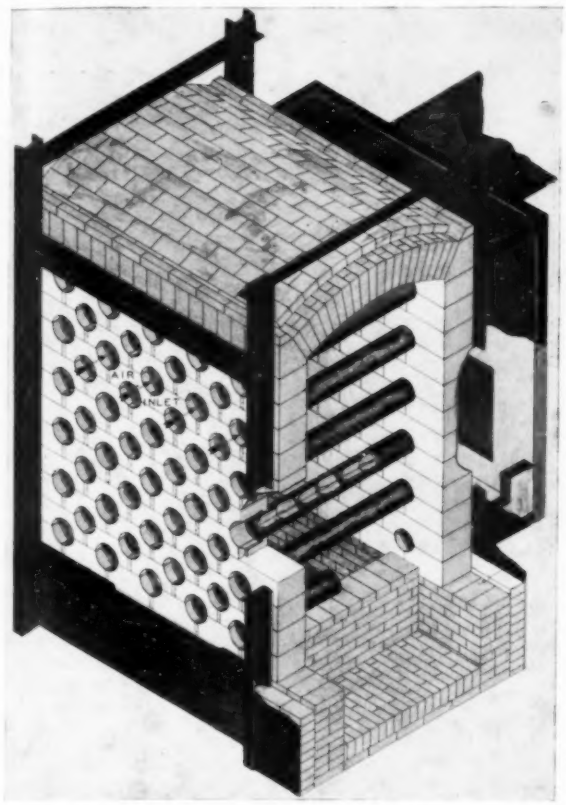


Fig. 2. Small standard recuperator unit.

6 in., the various sections being staggered or offset throughout the tube so as to break up the air flow and promote heat transfer in the manner shown by Fig. 2.

Savings

The direct savings to be effected in fuel by the use of such a recuperator to heat the air for combustion is readily calculated by combustion equations, and represents the heat put back into the furnace in the form of sensible heat in the combustion air, plus a factor for improved combustion conditions. In addition to this, however, preheated combustion air gives higher flame temperatures and a more rapid flame propagation, which is of particular benefit in low-temperature furnaces or with fuel of a low heating value such as producer gas. As the heating rate is to a great extent determined by the temperature difference between the heating medium and charge, the higher temperatures resulting from combustion with preheated air will result in a more rapid heat delivery to the charge, and the more lean the fuel the more marked is this effect upon the heating rate.

Other Uses

The advantages of this silicon-carbide bonded material for tubes are such that they are also being increasingly used for radiant tube furnaces. For such work, as the radiant tubes have frequently to be 12 ft. and 15 ft. in length to suit the size of the heating chamber to which they are applied, the limitations due to the length of the silicon-carbide tubes have been overcome by providing each end of the section with a lapped ball-and-socket joint which permits a certain misalignment without leakage.

Another development is the use of such tubes as hearth rollers, in which case the design comprises a water-cooled hollow steel shaft with the silicon-carbide tube or roller mounted concentrically and the intervening space filled with refractory insulating cement.

More recently still, silicon-carbide tubes are also used for external heating for the cracking and reforming of natural gas, propane, and butane. The material is not, however, suitable for use on the basic chemicals such as caustic soda and lime at high temperatures, while molten coal slags will readily dissolve it. Nor are these tubes satisfactory for use with sulphur vapours above about 900 °C., although when these are diluted with carbon monoxide no harm generally results.

A.D.A. Education Service

SINCE itsince ption the Aluminium Development Association has realised the importance of the educational aspects of its work and for several years now has been laying the foundation of a comprehensive educational service for technical colleges, secondary schools and industrial training establishments. Facilities have also been provided to assist the educational work of professional societies.

A special department of the Association was formed to cope with the increasing demand resulting from the rapid development of the aluminium industry and this department is now being expanded still further. In 1949 the services supplied included many lectures by the Association's staff, film shows to more than 15,000 students and the supply of literature and specimens for the use of teachers in hundreds of schools.

In the light of direct experience of the needs of teachers, visual and other teaching aids have been devised and the first of these, Wall Chart No. 1: "Aluminium Production—Mine to Metal," has recently been published. Supplied with the chart will be samples of raw materials and aluminium alloys. Future development of the scheme involves the publication of special educational information, further Wall Charts, film strips and the expansion of the film-lending library now firmly established.

Special exhibits are available on temporary loan to technical colleges and, where required, arrangements can be made for a discussion on the problems of giving instruction on aluminium technology.

At present, educational aids on this scale can only be made direct to schools and colleges on the application of Principals or Heads of Departments. They are supplied free of cost or of other obligation by the Aluminium Development Association at 33, Grosvenor Street, W.1.

MR. E. B. PIGGOTT has resigned from the position of Secretary of Newman Industries Ltd., for health reasons. MR. CHARLES BUSH, who previously held an Executive position with Messrs. Wimpey Ltd., has been appointed to succeed him.

CURRENT LITERATURE

Book Reviews

THE ROLLING OF METALS

Dr. L. R. Underwood, D.Sc., M.I.Mech.E. "The Rolling of Metals," 8vo, vol. 1, pp. xv + 344. Illustrated. London, 1950, Chapman & Hall, Ltd. (Price 42s.).

THE appearance of this book marks an important, though long overdue, development in the art of rolling metals in this country, because it is the first important English printed book dealing specifically with the theory of the metal rolling process, and the foreword has been contributed by the immediate past-president of the Iron and Steel Institute, viz., Sir Andrew McCance, D.Sc., F.R.S. The book had its origin, no doubt, in the excellent pioneer work done before and during the second great war by the Rolling Mill Committee of the Iron and Steel Industrial Research Council, because in 1939, in association with this Committee, Dr. Underwood began the arduous task of examining and collating the scattered literature related to the rolling of metals. In 1946, the Committee produced its First Report—I. and S. Inst. : Special Report No. 34—and Dr. Underwood's experience and his work in connection with this Report made him eminently qualified to write the book under review.

The book provides a highly informative and excellently critical survey of the theoretical and experimental investigations on the phenomena associated with the deformation of metal which is rolled between a pair of plain rolls, and the subject matter is almost wholly related to the hot and cold rolling of strip and sheet metal. A brief summary of chapters is given below : Chap. 1. The purpose of rolling research. Chap. 2. The forces acting in the region of contact between the material and the rolls ; Forward and backward slip ; The neutral point. Chap. 3. Detailed discussion of forward slip and the several factors related to it. Chap. 4. The flow of material in rolling, with reference to tests on ferrous and non-ferrous metals ; Methods of investigating flow ; Compression rate. Chap. 5. Yield stress and the criteria of yielding ; Theories of strength ; Hencky-Mises condition of plasticity ; Experimental methods of determining yield stress ; Homogeneous compression ; Factors affecting yield stress values. Chap. 6. External friction between the rolls and the material ; The function of friction ; The effects of rough and smooth rolls ; Methods of finding the co-efficients of roll face friction ; Variation of friction along the arc of contact. Chap. 7. Factors affecting the rolling load ; and the specific roll pressure with special reference to thin sheet and strip rolling ; The influence of roll flattening and other factors ; The effect of front and back tension ; Experimental investigations by Siebel and Lueg on the pressure distribution over the contact arc. Chap. 8. Theories of rolling applicable to thin sheet and strip ; The friction hill ; Investigations by von Karman, Nadai, Trinks, Tselikov, Orowan, Smith, Cook and Larke. Chap. 9. Use of the rolling theories for calculating the specific roll pressure, with and without strip tension ; Application of the theories to the hot rolling of sheet and strip. Chap. 10. Roll flattening and the calculation of specific roll pressure ; Hitchcock's investigations on roll-flattening ; The effect of various factors on roll-flattening.

The treatment is excellent throughout, and the book has the merit of discussion on a mathematical basis which is not too difficult to follow, and consequently the influence of various factors can be more correctly gauged. Careful consideration is given to the theories of rolling developed by von Karman, Nadai, Trinks, Tselikov and Orowan, and numerical calculations are included to illustrate the practical application of the theories. Their limitations are discussed, and comments and criticisms about them are valuable in suggesting where further investigations are required. On the matter of compression tests in relation to rolling, it is unfortunate that the important work done by Polakowski (I. and S. Inst. : J1, 1949, Vol. : 163), in this field was too recent to be available for discussion in Chap. 5. The metallurgical aspects of the deformation process have not been discussed, and the author advises readers to refer to the special researches which have been published in this country and elsewhere. A comprehensive classified bibliography, containing over 450 references, is included, and also a well compiled general index. The text is illustrated by 142 Figs., some of which facilitate the work of calculation.

On the side of criticism, it seems fair to suggest that the author has not adequately discussed the essential mechanics of the rolling mill as a deforming machine. There is no reference to single roll driving and its different effect on the process of deformation. The inclusion of the additional factor of roll neck friction is necessary when discussing the character of deformation in this type of rolling. Some of the Figs. could be improved for the sake of clarity in following the text, e.g., Fig. 1 in relation to the discussion on p. 13. There is a slight misprint in equation 10–16a on p. 294, and slight error of spelling and duplication occurs in references M.5 and M.24, pp. 336–7. The cine-camera technique proposed on p. 82 has been already applied by MacGregor and Coffin (J1, App., Mech., A.S.M.E., March, 1943). The true stress-strain curves, reproduced in Fig. 54, p. 122, were obtained by Siebel and Pomp in 1927, and Nadai (C.48) has only requoted them. In view of the customary meaning of "strain" in the theory of elasticity, it seems desirable to apply the term "Stress-deformation" or "Stress-elongation" to the curves shown in Fig. 54. The content of Fig. 42 was it seems, originally due to Rummel (Stahl und Eisen, 1919, Vol. 39). Except for the reference to Nadai's book, the classified bibliography does not contain any reference later than 1940 in the sections A to L, and Section M does not adequately cover investigations carried out from that date up to the time of publication.

The book provides an important introduction to the very large amount of investigation which has been carried out and which is being continued at an increasing rate, and those who are interested in rolling mill problems would find valuable information in the book. The promise of publication of a second volume, covering lateral spread, rolling torque, rolling load, power calculations, etc., will make Dr. Underwood's complete work

an outstanding contribution to the literature on the rolling of metals. The publishers have produced a fine book, excellent in both printing and binding.

J. S. CASWELL.

METALLURGICAL APPLICATIONS OF THE ELECTRON MICROSCOPE.

Published by the Institute of Metals, London. Cloth, 170 pp., 50 plates and numerous figures, 21s. or $3.50, post free.

THIS volume contains 13 papers presented at a Symposium held in London in November, 1949, a report of which appeared in the December, 1949 issue of *Metallurgia*. Some of the papers are of a review character and summarize the work that has been done so far in Great Britain, France, Germany and the United States; others record research results hitherto unpublished.

The Symposium was attended by about 300 scientists from various countries. The discussion, which is fully reported in the Monograph, contains important contributions from metallurgists and from leading workers in the field of electron microscopy.

The Monograph as a whole forms, therefore, a comprehensive and up-to-date guide to the use of the electron microscope in metallurgy and, besides being valuable to all physicists and metallurgists already working in the field, it will serve as a most useful introduction to the subject for those metallurgists who may not yet fully appreciate the potentialities and limitations of this comparatively new technique.

THE PROPERTIES OE METALLIC MATERIAL AT LOW TEMPERATURES

By P. Litherland Teed. Volume I. of a series of Monographs on Metallic Materials, published under the authority of the Royal Aeronautical Society 8vo. Pp. viii+ 22. 1950, London: Chapman and Hall Ltd. (21s. net).

IN the regions of the upper air (above 35,000 feet), the natural habitat of the modern aircraft, the mean atmospheric temperature is — 56·6°C.; and sometimes some 35°C. lower. This new factor makes the properties of metals at low temperatures of extreme importance to the aircraft designer, although many new industrial refrigeration processes e.g. the production of butyl rubber, the freeze-drying of penicillin, etc., have given the chemical engineer also problems in finding suitable materials of construction for his plant. These considerations prompted the Royal Aeronautical Society in their sponsoring of the present volume, an opening shot in their new series of monographs on metallic materials.

Major Teed is an authority on metals for use in aircraft, and in his present task he has had the co-operation of a government department and a number of firms, as well as three development associations and many private individuals of high repute. For all that, and it is no fault of Major Teed's this book cannot be said to be any thing other than disappointing, especially, one would imagine, to its designated public the aircraft industry. Disappointing, mainly because the experimental work reviewed by the author is far from complete; work in this field, with a few notable exceptions, has been scanty and sketchy and suffers in many cases from the common disability of insufficient indentification, by careful analysis, of the metals concerned. However, a safe conclusion from this book is that nearly all metallic materials improve in physical properties with decreasing temperature, the only important exceptions being the impact value of certain steels, zinc alloys and tin alloys and the allotropic modification of tin.

The author seems to have made an exhaustive search of the literature, from which he quotes extensively, and the addition of certain information from several firms produces a total of about 100 pages of tabular matter in the book. This mass of information the author marshals skilfully, and there seems little doubt that he has done full justice to a subject which hardly does as much for him. A very useful feature is a section at the end of each chapter summarising concisely the lessons on each material discussed, as far as firm conclusions can be reached; Major Teed's main conclusion is that "a vast area still remains uncharted, unknown, yet waiting to be explored." But the book can be recommended as authoritative as far as it goes.

It is well produced, with an adequate index, but it is difficult to approve the system adopted for denoting references and presenting footnotes. It might also be mentioned that on page 9 there is a table which does not include the corresponding reference numbers of the figure to which it refers.

W. G. ASKEW.

STATISTIQUES

By Mierais et Métaux, S.A., 55, Rue d'Amsterdam, Paris 8e, 1949. pp. 160.

THIS book covers the non-ferrous metals copper, lead, zinc, tin, antimony, cadmium, cobalt, nickel, aluminium, magnesium, silver and gold, from 1939. It deals with the production, imports and exports of the principal countries, and summarises world production, consumption and prices in text and tables. Many coloured maps indicate the main mineral deposits and refineries and the book concludes with the commodity quotations of London from 1913 to 1948. This collection of metal statistics thus provides a set of most valuable retrospective data.

F.M.

WELDING DICTIONARY

French — German — Spanish — English. Compiled and edited by R. N. Thompson, editor of *Welding*, and G. Haim. Published by The Louis Cassier Co., Ltd., and distributed by Iliffe & Sons, Ltd., size C8vo ($7\frac{1}{2}$ × 5 in.), 234 pages. Bound in imitation leather with jacket. Price 21s. (postage 6d.).

Like all new techniques, welding has given rise to a considerable number of new words and phrases which have specialised meanings. This leads to difficulties in the reading of foreign literature because these new welding words and expressions are not to be found in the dictionaries so far available.

The purpose of this dictionary is, therefore, to enable the welding engineer and research worker with an average knowledge of another language beside his own to be able to understand easily the technical publications of a different country.

In addition to terms specifically related to welding, many other expressions pertaining to electrical and mechanical engineering and to metallurgy are included, as the ready translation of such terms are essential for the appreciation of any welding literature.

The dictionary has been divided into four parts—French, German, Spanish and English—the English part being treated as a key glossary with equivalents in the other three languages. Fourteen pages of tables are included which add to the usefulness of the dictionary.

W. A.

METAL STATISTICS, 1950.

By N. J. Langer, pp. 800 (The American Metal Market, New York). 82.

THE 43rd annual edition of *Metal Statistics* contains the same general assortment of statistical information, on ferrous and non-ferrous metals and miscellaneous economic subjects, as previous issues. Due to the continued absence of reliable data from behind the Iron Curtain, world statistics on production and consumption remain unsatisfactory as far as totals are concerned. Consequently, where such totals are shown, they are still largely conjectural.

As in all previous editions, the prices are generally based on the quotations published in The American Metal Market, and represent wholesale prices. Statistics dealing with production and consumption are gathered from various authentic sources, in particular from the American Iron and Steel Institute, the American Bureau of Metal Statistics, the U.S. Bureax of Mines, the Bureau of the Census, the Copper Institute, the Lead Industries Association, and the International Tin Study Group.

A buyers' directory of some 130 pages, arranged alphabetically according to products, will be of considerable value to industry. F.N.

Trade Publications

WIGGIN NICKEL ALLOYS

THE new issue of the above publication (No. 327) is largely devoted to the use of high nickel alloys in aircraft construction. Illustrated articles feature Wiggin thermometals in aircraft altimeters, Monel in the Airspeed " Ambassador " and in the Westland-Sikorski helicopter, Inconel in aircraft engine exhaust systems, nickel-iron alloys in fire detector equipment, thermal de-icing of aircraft windscreens using nickel-chromium resistance wire and the use of nickel-chromium and nickel-copper alloys in electrically-heated covers for oil tanks and pipelines. In addition, the publication covers some uses of nickel-containing materials in automobile engines, notably the K.L.G. plug. Copies of this publication may be obtained, free of charge, upon application to : Henry Wiggin & Co., Ltd., Wiggin Street, Birmingham, 16.

NOTES ON THE HEAT-TREATMENT OF ALUMINIUM ALLOY CASTINGS

THIS latest technical data sheet, issued by Alar, Ltd., lists the heat-treatment conditions for all the heat-treatable alloys included in B.S. 1490, and contains notes on recommended practice, the heat treatment of pressure die castings, and stabilising or stress relieving.

A 64-page booklet has been received from George Kent Ltd., Luton, Bedfordshire, illustrating and describing in detail their range of industrial instruments and controllers. It has the Reference No. 1002 and is in French. A similar edition is in Italian and we are advised that shortly similar editions will be available in Danish, Swedish and German. The full range of this Company's instruments is described in a lucid and useful manner and the booklet, which is well produced and bound in stiff covers, will be especially useful to all users of industrial instruments that are concerned with the measurement and control of gases and liquids and of temperatures.

A Reference Stoving Paint for Corrosion Tests

A DIFFICULTY in carrying out laboratory tests on protective schemes for iron and steel has always been the absence of generally accepted standards of comparison. The British Iron and Steel Research Association's Methods of Testing (Corrosion) Sub-Committee have partly met this difficulty by preparing a reference stoving paint, which comes within specification CS 2249. As a standard basis of comparison it is likely to be of use to all those concerned with exposure and other tests of protective schemes for iron and steel.

The formulation is obtainable from B.I.S.R.A. Corrosion Laboratory, 514, Bristol Road, Birmingham, 29. Samples of the paint are also available from the same source at a small charge.

STRUCTURAL and Mechanical Development Engineers Ltd. (known as S.M.D.), of 2, Buckingham Avenue, Slough, announce that the Air Ministry, acting on behalf of the Ministry of Civil Aviation, has placed an order on them for the design, construction and erection of a three-bay hangar for London Airport, to be manufactured entirely in aluminium alloy ; each bay 150 ft. overall span width by 110 ft. long, with clear doorway openings of 125 ft. × 30 ft. high.

British Rolling Plant for Norway

A VERY considerable increase in iron and steel production will result when the new integrated plant of A/S Norsk Jernverk goes into production in 1954. Situated at Mo-i-Rana, 25 miles south of the Artic Circle, it represents the fulfilment of plans of many years standing to develop an enlarged steel industry in Norway, based on her own natural resources in the shape of local ores and hydro-electric power.

In the face of determined German competition, Davy and United Engineering Co. Ltd. have secured an order for rolling mill equipment worth more than a milliion pounds, which will include a high-lift reversing blooming and slabbing mill, a two-high reversing three-stand structural mill and auxiliary equipment. Handling bloom ingots up to 3½ tons in weight and 5 ton slab ingots, the inital output of the plant will be up to 220,000 ingot tons per year, although the blooming mill is being designed for a total capacity of 650,000 tons per year against future expansion plans for the works.

The blooming mill, 42 in. × 9 ft. with a 58 in. lift will be powered by a twin motor drive and will have electric universal manipulators, twin motor screwdown, hydraulic balance on top roll and both spindles, and housings weigh about 70 tons each. The electric bloom shear will cut up to 12 in. × 12 in. blooms or 36 in. × 5in. slabs and will be followed by a bloom transfer for sale blooms and light slabs. Provision is made in the approach table to the first stand of the structural mill for transfer to a waste heat furnace for reheating.

The three-stand structural mill will be driven from one end through pinion housing and spindles by a single electric motor drive. The first stand is a 32 in. mill, the remaining stands being 30 in. mills. Billets up to 6 in. square coming from the second stand can be sheared by a 350 ton electric billet shear and either kicked off into cradles or carried over a cooling bank for eventual sale. Products smaller than 6 in. square will be transferred to the third stand, run straight out, sawn to length, and passed to the cooling bank.

High Temperature Hardness Testing

Some Recent Russian Work

Recent Russian work on hardness testing at high temperatures has been directed towards obtaining information on the high-temperature properties of alloys more rapidly than is possible by the usual methods. A Brinell machine fitted with a metallo-ceramic indentor has been used for this purpose.

A NEW method of hardness testing alloys at high temperatures has been developed recently in the All Union Institute for Aviation Materials, and the view elaborated that such hardness tests give much more reliable indications of the true qualities of the alloys in question than those at ordinary temperatures. A special method of protecting the test-pieces from oxidation, and for heating up to 900° C. is included. Moreover, a close relationship is said to have been discovered between high temperature hardness and what is described as long term stability. Results have been reported by I. L. Mirkin, *et al.* (*Zavodskaya Lab.*, 1949, No. 9, pp. 1,080–1,087.)

It is pointed out that the most usual method for determining the heat resistance of alloys at high temperatures is through a study of creep and other factors in stability. This consumes much time and material, however, and simpler and cheaper methods are needed. It is claimed that these are to be found in high temperature hardness tests, which may serve as reliable indicators of other characteristics, such as ageing effects, yield point, etc. Other references include Ya. B. Friedman, "On the mechanical properties of metals," *Oborongiz*, 1946 (State Bookshop—Defence Dept.), and A. A. Bochvar, *Izvest. Akad. Nauk, S.S.S.R.*, ONT, 1947, **10**. The latter, in work on hardness in relation to creep, recommended indentation at moderately high temperatures, up to 300° C., for light alloys. The usual method of testing hardness with the Brinell apparatus can only be used conveniently for temperatures not exceeding 500° C., with either diamond or Pobedite indentors. In either case these must be protected from oxidation or cracking and to this end vacuum or inert atmospheres have been proposed—e.g., by Bence (*Amer. Soc. for Metals*, 1948, **38**, 505); but this is troublesome and complicates working.

The apparatus used in the present research on high temperature hardness was a Brinell machine, with metallo-ceramic (ceramet) indentor, for temperatures up to 900°, without vacuum or protective media. Risk of oxidation of test-pieces under these conditions was considered negligible. In order to protect the indentor, however, a special chemico-thermal diffusion treatment of the ball was evolved, of which no further details are given, except that a protective film was formed on the ball. In the original, photographs showing the difference between treated and untreated Pobedite are displayed. A ball of 5 mm. diameter was used at a load of 750 kg. At the Institute of Metal Bearings, the Pobedite balls were ground to an accuracy within a few microns. The portable apparatus set-up is shown in the original, both in a general view and in diagrammatic section. Briefly it is an ordinary Brinell tester to which has been added a vertical tubular electric heating furnace with thermo-couples, etc. It was used in the ordinary way and the test pieces were cooled down to room temperature for measurement of the impressions. The possibility that, on cooling, the test-pieces would contract and yield misleading values as to size of impression was, in the author's view, excluded; in cooling from 800° to 20° C. a 2 mm. diameter impression was reduced by only 0·02 mm. It should be added that the period of test was 30 sec., and the foregoing statement on loads should be qualified to the extent that, for hardnesses below about 300 H_B 750 kg. was used, and above that hardness figure the load was 250 kg. in order to preserve the indentor.

Tests with heat resistant alloys are described in some detail, beginning with the individual basic metals used in making the alloys; also solid solutions prepared with these metals. The basic metals were tungsten, molybdenum, chromium, cobalt, nickel and iron. The fall in hardness as temperatures increase to 800° C. is presented graphically for these and also for alloys based on the last four—i.e., those prepared by casting. Tungsten and molybdenum alloys were prepared by powder metallurgy methods (sintering, etc.). The chromium alloys were by far the hardest at temperatures up to 800° C. (335 H_B), whilst those based on iron had, at this temperature, hardness of only 100 H_B, with the nickel and cobalt alloys slightly higher (140 and 180 respectively). The alloys group themselves in the same relative order as the basic metals, but there is a much wider gap between chromium alloys and the rest, than is the case with the pure metals. With chromium alloys, the hardness at room temp. is 470 H_B, whilst iron, nickel and cobalt alloys respectively are 210, 250 and 270 H_B, and they all lose approximately 120 points at 800° C.

The "hot" hardness tests become still more interesting if the results are compared with those obtained by standard methods, such as the time required for failure under tensile stress at constant load. It is shown that the alloys again group themselves in the same order. The hot method, too, is useful in judging the effect of stabilising additions to the alloys—small percentages of different elements—although no details are given as to such additions. A nickel-chromium iron alloy, at 800° C., had hardness of only 70 H_B, and addition of cobalt, even up to 20%, did not raise it above 74. The times for failure under load of these solid solutions were of the same order. At 800° C., and a load of 5 kg./mm.², period for nickel-chromium-iron was 76 hr., and for nickel-chromium-iron-cobalt, 98 hr. Addition of alloying elements and suitable heat treatment or ageing considerably increased high temperature hardness, from two to four times. It is again emphasised that the "hot" hardness tests are much more reliable in estimating the effect of additional constituents, and/or heat treatment, than measurements at room temperature, which may yield misleading results.

Still more important is the relationship between these hardness tests at high temperature and long-term heat resistance or stability. This is exemplified in the case of high-nickel alloys and graphs show a linear correlation, with time in hours as abscissae and hardness as ordinates. Other factors, of course, also enter, not necessarily related to hardness, as a result of which, for example, previously insignificant plastic deformation may lead to collapse and fracture; then the relationship between stability and hardness may break down.

The next point considered is the time factor in relation to indentor load, as recommended by A. A. Bochvar (*loc. cit.*), who stated that the fall in hardness values in a given time period should be characteristic of creep in metals at the given temperature. In testing "long period" hardness, with gradually increasing time of load application, there should be a diminution in stress owing to the steady increase in the diameter of the impression. This in turn should lead to a gradual slowing up of the fall in hardness, according to the nature of the metal or alloy. The ratio of the diameter of the impression to that of the ball in course of time decreases, and thus also the "condition of similarity" no longer obtains. The present authors chose experimental conditions in which the relationship between the diameter of the impression obtained (2–3 mm.) and the ball diameter (5 mm.) was of the order of 0·4–0·6, that is to say, not exceeding the limits usual in hardness tests.

V. P. Shishokin found that between the diameter of the impression and the time of load application there was a definite relationship (*J. Techn. Phys.*, 1938, **8**, 1,613–1,628). From this the present authors have developed what they call their method of "three short hot hardness tests," in which each test is ten times as long as the preceding one. The first is for 30 sec., the second for 5 min. and the third 50 min. It is not considered necessary to extend the tests to eight hours, since the fall in hardness is hardly appreciable after a certain time. Results of tests with high-nickel alloys, using the "three-short-hot" method, plotted in the original, confirm this, and show very close agreement with those of prolonged tests. The following figures are given for some nickel alloys, Nos. 32–34 described as "complex alloyed" or modified:

Alloy	Heat resistance or stability, time in hours for fracture	Hardness H_B for times of load application		
		30 sec.	5 min.	50 min.
No. 32	35	229	198	160
No. 33	60	248	217	179
No. 34	160	235	207	193

The authors' comment on this, that No. 32 gives the highest hardness figures as a result of ageing treatment at 600–900° C., is not very clear. They add that hot hardness tests sometimes reverse the results of those at room temp. A diagram shows that alloys Nos. 1 and 2 at 20° C. have about the same hardness figure of 270, with No. 1 slightly harder than No. 2. At 800° C. the position is reversed, and No. 2 has 220 and No. 1 has 190 H_B. Alloys Nos. 3 and 4 have a substantially different hardness number at 20° C., but the decline in hardness of both is much greater at 800° C. than with Nos. 1 and 2; their position is again reversed (No. 3 370 H_B and No. 4 110 H_B). Their composition is not disclosed.

Staff Changes and Appointments

Dr. R. T. Parker appointed Director of Research and Mr. George Forrest, Associate Director of Research of Aluminium Laboratories Limited. Dr. Parker succeeds Mr. G. H. Field, C.B.E., who has retired from active service.

Mr. Paul Parfitt, A.I.M., has resigned his post of Chief Metallurgist to Messrs. Turner Manufacturing Company, Ltd., Wolverhampton, to take up an appointment of Chief Metallurgist to Messrs. John Fowler and Company (Leeds) Ltd., Leeds. The Company is a member of the Marshall Group.

Mr. R. H. Cooke of Philips Electrical Ltd., has been appointed Assistant Manager of the X-ray Department. His appointment took effect from July 1st, 1950.

Mr. C. L. G. Fairfield, M.A., A.M.I.E.E., A.M.I. Mech.E., has been made a Director of Mullard Equipment Ltd. The appointment takes effect from August 1st, 1950.

Mr. G. Smellie has been appointed Works Manager of the Margam, Port Talbot and Abbey Works, of the Steel Company of Wales Ltd., with a view to centralising the administration of the Steel Division with effect from the 1st September, 1950. As Works Manager of the Port Talbot and Margam Works, he has, during recent years, had the difficult task of maintaining the production of these works in spite of all the reconstruction work which has been going on at the same time, and it is interesting to note that during this period every department in these works has steadily increased their output figures.

Nimonic 90 for Gas Turbine Blades

The Mond Nickel Co., Ltd., advise us that they are now able to announce the addition of Nimonic 90 to the published Nimonic series of alloys. Although this superior material has already been installed on many gas turbines, its use has been restricted and details of its properties have not so far been available for publication owing to the fact that it has been under a security ban.

Nimonic 90 results from extensive research in our laboratories, combined with long production experience in the rolling mills of Henry Wiggin & Co., Ltd. Many service trials in gas turbines have demonstrated its superiority as the material for gas turbine blades. At 750° C. it is 10% better than Nimonic 80A under all conditions. At 815° C. and even at 870° C. it has shown high load-carrying capacity for long periods.

It is available in bar, sections, forgings, sheet strip and wire.

Symposium on High Temperature Steels

The Council of The Iron and Steel Institute have reluctantly decided that the above Symposium must be postponed, as the recent dispute in the Printing Trade has made it impossible to print advance copies of the papers for discussion in time for the meeting. The Memorial Lecture is likewise being postponed.

The new dates are not yet known, but a notice will be issued in due course when all details for the later meeting and Lecture have been settled.

Notices of this postponement are being sent to the authors of papers and to all who intended to be present at the meeting and Lecture.

Recent Developments in Materials, Tools and Equipment

The Murex Type H500 Electrode Holder

THE Murex Type H500 electrode holder has been specially designed for heavy duty electric arc welding and has many outstanding features. It has special long life jaws and is all-insulated. All parts are replaceable with spares, the holder is light in weight, (24 oz.) excellently balanced, robust in construction and it complies with B.S.S. No. 638

Suitable for currents from 100 to 500 amperes, it can be used for all electrodes from No. 8 gauge to $\frac{3}{8}$ in. dia. and its jaws are specially grooved to facilitate automatic positioning of the electrodes. The jaws are made of a special alloy having a very high resistance to burning and spatter adherance. They combine strength with current carrying capacity ensuring long life.

All live parts of the holder are shrouded in special shock-proof insulating material which has been developed as a result of considerable research. The insulation is tough and will successfully withstand intense heat from the arc for prolonged periods. The insulated parts are of generous proportions, the hand being well protected by a circular guard.

All parts of the holder may be replaced and interchanged with spares which can be supplied seperately if required. These interchangeable spares provide the maximum economy as it is not necessary to replace the complete holder for the sake of one part. The holder is assembled or dismantled by the simple Allen key supplied with it and no engineering skill is required in the assembly, Allan screws being used throughout.

Murex Welding Processes Ltd., Hertford Road, Waltham Cross, Herts.

Wire Rope Parting Machine

A NOTABLE contribution to wire rope practices is made by British Insulated Callender's Cables Ltd., by the introduction of a new wire rope parting machine. The first of its kind to be marketed, the machine parts wire ropes in such a manner that the ends are sealed and left in a tapered form, thereby greatly facilitating threading through blocks or pulleys and subsequent clamping. This BICC development, therefore, will be of great assistance to manufacturers and stockists of wire rope, in that it obviates binding and cutting by conventional methods and eliminates fraying of wire ends or the string wrapping finish employed hitherto.

The machine is suitable for steel and hemp cored ropes ranging from $\frac{3}{8}$-inch diameter to $\frac{7}{8}$-inch diameter, but has an occasional use capacity for parting of ropes up to $1\frac{1}{8}$-inches diameter. The parting operation, which is both simple and remarkably fast, may be briefly described as follows.

The required length of rope is first measured off. The machine is then set to accommodate the size of rope to give the desired length of taper, and to operate at the correct speed. These adjustments having been made, the rope is lowered into the machine and clamped in the contact grips provided. A heavy electrical current of low voltage is then passed through a portion of the wire rope held between the clamping heads, thus heating it to such a degree that it becomes malleable. In the initial heating stage, the rope is twisted to bring all the strands into close contact with one another and to ensure uniform heating throughout. Tension is then applied and the rope is stretched until a pronounced waisted effect is obtained. The effect of burning through is to allow the molten metal to weld the ends of the stands and wires securely together forming an attractive seal which prevents strands unlaying.

The length of time for which the current is switched on depends upon the size of the wire rope to be parted, and varies from about 45 seconds for a $\frac{3}{8}$ in. diameter rope to 2 minutes for a 1 in. diameter rope. These times compare with between 12 and 20 minutes required for the parting process at present in general use.

Delivery of this new machine is approximately ten weeks after receipt of order.

British Insulated Callender's Cables Ltd., Prescot Works, Prescot, Lancs.

An Ingenious Method of Guarding Four Sides of a Press

EARLIER this year, Radiovisor Parent Ltd., announced the introduction of a foolproof photo-electric press guard incorporating revolutionary design features. Briefly, the system consists of projecting a "curtain" of light forming a barrier across the opening of the press. The light falls on a "receiver" at the side of the machine and is concentrated on a number of photo-electric cells, the receiver being connected to a control unit incorporating relays which remain energised as long as the light rays are uninterrupted. Should, however the operator interrupt the barrier by moving his hand or any part of his body towards the danger area, the relays are de-energised, immediately stopping the press and making further operation impossible even if the machine controls are depressed.

Such a system, of course, protects only the front of the press which, in most instances, is quite adequate as the ends can usually be enclosed with ordinary fixed guards without any loss of efficiency. Certain types of machines, however, are open on two or more sides, as in the case of hydraulic presses, which generally require protection on all four sides. This problem arose recently when it was required to guard an hydraulic press measuring 10 ft. 8 in. long by 4 ft. 3 in. deep, and a very ingenious scheme was adopted to avoid the necessity for employing four separate units *i.e.* one for each side.

The front of the machine is guarded by a unit situated at the right hand corner: this projects a 16 in. deep by 2 in. wide barrier of light across the opening, the depth of the barrier being sufficient to provide an ample margin of safety when the press is opened to its maximum position The light falls onto a $16\frac{1}{2}$ in. by $4\frac{1}{4}$ in. mirror pedestal-mounted in a pressed steel housing situated at the front lefthand corner of the press, the housing being adjustable to permit alignment with the

rays. The mirror is fixed at 45° to the direction of the beam and, consequently, the light is reflected along the side of the press, falling onto a receiver mounted at the rear lefthand corner.

A second unit projects a light barrier along the opposite side of the machine onto another mirror set at 45°, this reflecting the beam across the rear to a second receiver. By this means, all four sides are fully protected by two units. Interruption of any beam causes the press mechanism instantly to stop, thus rendering the machine foolproof and safe even if any of the operators are hidden from the man at the controls.

Radiovisor Parent Ltd., 1, *Stanhope St., London, N.W.*1.

New H.F. Heating Generator

A NEW 2 kW. induction heater is announced by The General Electric Co. Ltd., for a wide range of applications including the hardening of small parts and particularly for soldering and brazing, where large savings in time and money can be effected. Special features are incorporated to permit the use of easily constructed heating coils and their quick interchangeability makes the equipment eminently suitable as a general purpose tool for processing batches of different articles.

Output control is obtained by means of a single control knob which adjusts the impedance matching of the load. By this method the correct operating conditions for maximum valve life and efficiency are maintained at all times.

Connection of the water cooled heating coil is made by means of a special coil block which can be adjusted to extend up to 5 inches from the side of the set to suit the work in hand or to facilitate the use of conveyors, handling gear etc. The coil operates at low voltage and is not therefore a source of danger.

Determined effort has been made to reduce to a minimum, the overall running costs of the generator. The oscillator circuit uses a single oscillator valve of well proved reliability and this valve is protected from damage due to mains fluctuations, by a voltage stabiliser in its filament supply.

The generator is of robust construction and is mounted on larger rubber-tyred castors. Supports for a work table are fitted to the side and, in keeping with modern machine shop practice, the set is attractively finished in green and cream.

The generator is supplied for operation on 360/440 volts 3-phase supply.

*The General Electric Co. Ltd., Magnet House, Kingsway, London, W.C.*2.

Vertical Broaching Machines

THE latest S.R.V. and D.R.V. ranges of British built vertical broaching machines by the Lapointe Machine Tool Co. Ltd. are noteworthy. The S.R.V. is a single-ram variable speed hydraulic surface broaching machine available in various sizes up to 25-ton capacity and 90-in. stroke. It incorporates the Company's latest design improvements, including their new low-pressure hydraulic system which possesses important advantages. All controls are conveniently grouped together, and the provision of dual push buttons which must be operated together prevents chances of injury. The ram speed may be changed instantly and smoothly simply by rotation of a single dial; constant lubrication of all moving parts is provided by a pressure system incorporating a safety device which automatically stops the machine should the oil level fall below normal.

All motions of the ram and fixture are electrically and hydraulically interlocked to ensure correct operation and sequence. For setting purposes, the interlock system may be by-passed, allowing the parts to move independently, the design being such that the machine must be returned to interlock control before it will operate on production runs.

The D.R.V., a double-ram machine of very similar design, is built in standard sizes up to 32-ton capacity and 66-in. stroke. A double tip-down fixture, useful for a very wide range of work, is included as standard equipment. Both models are essentially high-production machines.

The Lapointe Machine Tool Co., Ltd., Edgeware, Middlesex.

Liverpool Metallurgical Society

Student Paper Competition

IN connection with its activities for the 1950-51 session, the Liverpool Metallurgical Society has instituted a Student Paper Competition open to all, living or working in the Mersey or Deeside areas, who are engaged upon the full-time study of metallurgiy or who have not attained the age of 25 on October 5, 1950.

There will be two classes in the competition, covering papers dealing respectively with (a) A critical review of a subject within the field of physical metallurgy, and (b) A reasoned account of a process in the fields of metallurgical extraction or fabrication, for each of which a prize of three guineas is offered. Entries, in Typescript and of about 3000 words in length, should reach the Hon. Secretary, J. A. Pardoe, Esq., 183, Queens Drive, Liverpool, 4, by February 1, 1951.

The contributors of the prize-winning papers will be expected to present them at the Annual General Meeting of the Society to be held on April 5, 1951.

Head Wrightson and Co. Ltd., Thornaby-on-Tees, announce that the name of their subsidary company engaged in light alloy engineering has been changed from Head Wrightson Aldean Ltd., to Head Wrightson Aluminium Ltd. MR. R. B. W. BOLLAND has been appointed Commercial Manager and MR. R. H. STURGES Director and General Manager. SIR JOHN WRIGHTSON, BT. is Chairman.

Head Wrightson Aluminium Ltd.

British and American Steel Castings

IT has been announced that a basis of collaboration has been established between the Technical and Research Division of the Steel Founders' Society of America and the Research and Development Division of the British Steel Founders' Association.

It is understood that these two research organisations which have been formed and are strongly supported by the steel foundry industries in the U.S.A. and in the United Kingdom respectively—will exchange information relating to their research programmes and upon certain specified fields of research, both fundamental and applied.

CAMBRIDGE TEMPERATURE CONTROLLERS

ELECTRICALLY OPERATED

INDICATING CONTROLLERS
INDICATING ZONE CONTROLLERS
INDICATING ALARMS
RECORDING CONTROLLERS
POTENTIOMETER CONTROLLERS
POTENTIOMETER ZONE CONTROLLERS
TIME-TEMPERATURE CONTROLLERS
HEAT SENSITIVE ELEMENTS
AUTOMATIC VALVE GEAR, etc.

MECHANICALLY OPERATED

DIAL REGULATOR
ALARM INDICATOR
THERMOMETER REGULATOR
RECORDING REGULATOR
HYDROSTATIC RECORDING REGULATOR
PNEUMATIC REGULATOR
HUMIDITY REGULATOR, etc.

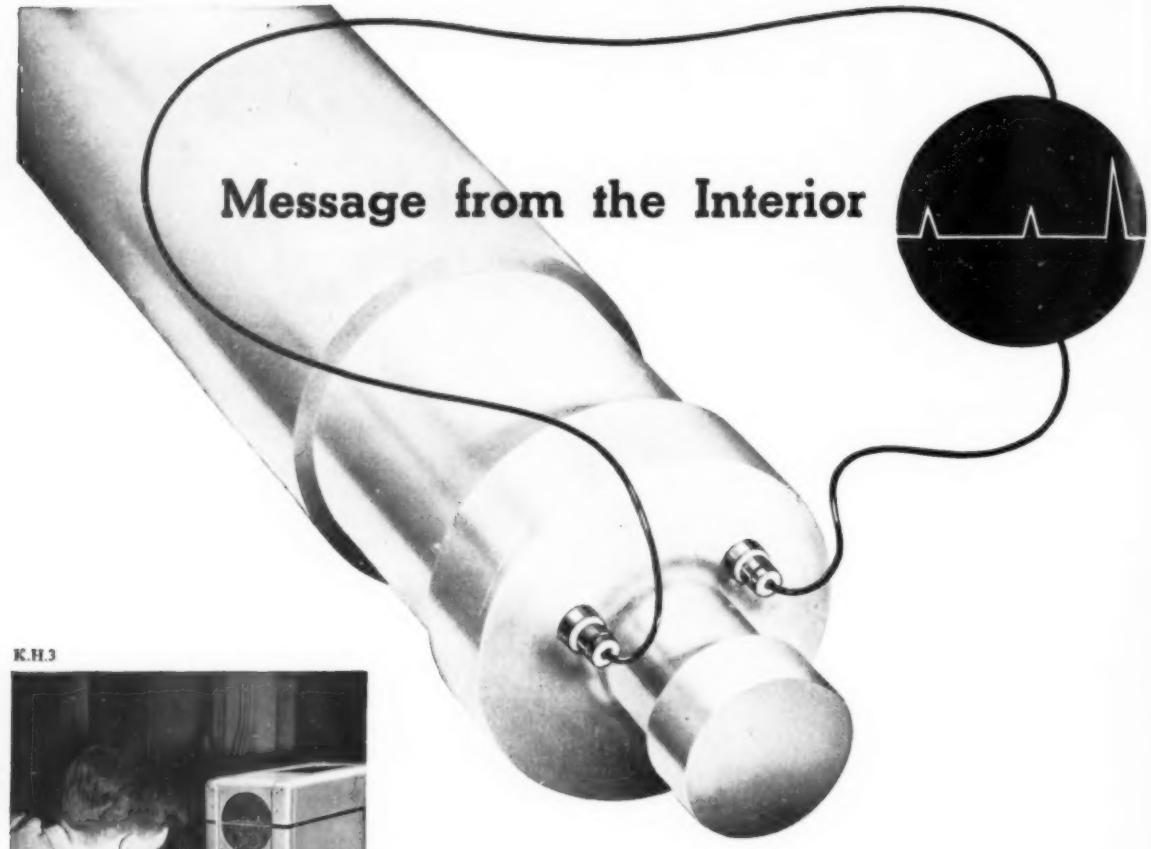
THESE USEFUL FOLDERS

Nos. 67v and 68v

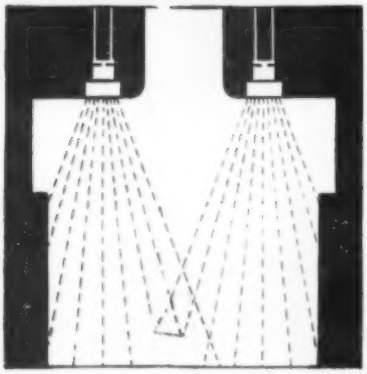
provide the latest information on a wide series of Automatic Regulators, from the simplest devices for controlling a pan or oven, to the most elaborate outfits for precise control in metallurgical applications. The instruments described cover the entire field of temperature control, by the operation of valves, levers, switches, contactors, or other mechanism, and may be applied to any temperature up to 1700° centigrade. Either or both Folders will be gladly sent post free on application

CAMBRIDGE INSTRUMENT COMPANY LTD
13, GROSVENOR PLACE, LONDON, S.W.1

Message from the Interior



K.H.3



THE HUGHES Mk. 4 SUPERSONIC FLAW DETECTOR enables flaws in metal—whether in billet form or as a finished component—to be detected unerringly without damaging the metal itself. Many cracks difficult to detect by radiographic methods are easily found. Moreover the equipment is portable, easy to operate and indicates the actual position of the flaws and not merely their presence.

A narrow beam of supersonic energy is directed into the metal under inspection by a small probe (the transmitter). Energy reflected by any flaws in the path of the beam and from the opposite boundary of the material is picked up by a second probe (the receiver) and its presence shows on a cathode ray tube, which thus indicates the relative positions of flaw and material boundary.

KELVIN HUGHES
PRECISION INSTRUMENTS

KELVIN & HUGHES (INDUSTRIAL) LTD., 2, CAXTON STREET, LONDON, S.W.1

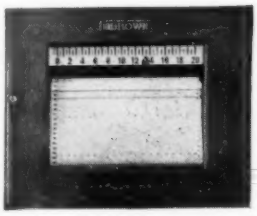
BROWN *Electronik*

continuous balance

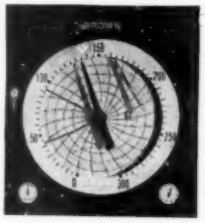
POTENTIOMETER

gives advanced temperature indication recording and control

OVER 50,000 INSTALLATIONS THROUGHOUT THE WORLD



Brown "Electronik" Single or Multi-point Strip Chart Potentiometer

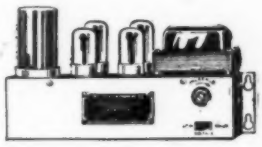


Brown "Electronik" Circular Chart Potentiometer Recorder Controller

Brown "Electronik" Single or Multi-point Precison Indicator. Self-Balancing with integrally mounted push-button switches

BROWN "Electronik" Potentiometer pyrometers, by replacing conventional galvanometer and cyclic balance mechanisms with a simple self-contained electronic amplifier assembly, provide continuous measurement and high sensitivity. Simplicity and ruggedness of construction permit great reduction in maintenance costs and reduce "time off-process" to a minimum. Please write for specification bulletins.

CONTROL On-off, proportional and integral action available for either electric or air operation. Derivative action also available with air operation.



Amplifier Unit

Now available from British production

HONEYWELL

Advanced FOR BETTER HB PROCESSING *Instrumentation*

BROWN

HONEYWELL-BROWN LIMITED, 1 Wadsworth Rd., Perivale, Greenford, Middx. Works: Blantyre, Lanark, Scotland

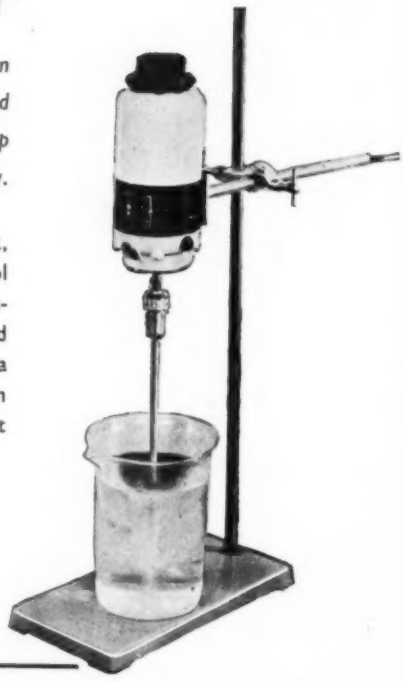
Affiliated companies: Stockholm, Brussels, Amsterdam, Zurich.

From Stock–

Universal laboratory Stirrer

The Griffin and Tatlock Laboratory Stirrer performs any stirring operation simply, quietly and efficiently. The readily interchangeable direct and flexible drives, the built-in speed control and the vat or drum clamp comprise a versatile, universal unit indispensable in any laboratory.

SPECIFICATION. The AC/DC power unit, to which an integral toroidal speed control is fitted, gives speeds up to a no-load maximum of 5,000 r.p.m. The flexible drive and special drum clamp are accessories, while a grooved pulley on the motor spindle can give a belt drive if necessary. Leaflet GT 1388/43 free on application.



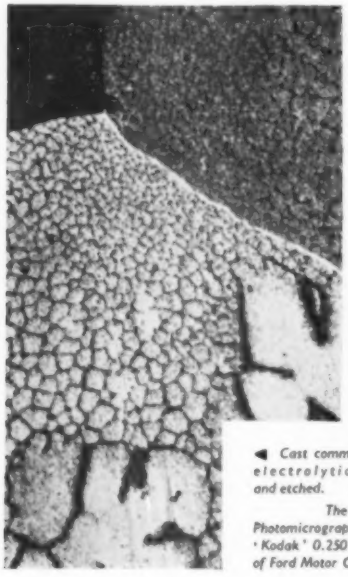
GRIFFIN and TATLOCK Ltd

Established as Scientific Instrument Makers in 1826

LONDON Kemble St., W.C.2 — **MANCHESTER** 19 Cheetham Hill Rd., 4 — **GLASGOW** 45 Renfrew St., C.2 — **EDINBURGH** 8 Johnston Terrace, 1

BIRMINGHAM: STANLEY BELCHER & MASON LTD., Church Street, 3

0.250 –the plate for PHOTOMICROGRAPHY



◀ *Cast commercially pure lead, electrolytically polished and etched. X25.*

The same, X1000. ▶ *Photomicrographs made on 'Kodak' 0.250 Plates. (Courtesy of Ford Motor Co. Ltd.).*

'KODAK' 0.250 METALLOGRAPHIC PLATE has a fine-grain high-contrast emulsion of high resolving power, especially colour-sensitized for photomicrography and metallography. It has its highest sensitivity in the region of maximum visual sensitivity, where most microscopes yield sharpest focus, and comparatively little speed is therefore lost when working with a green or yellow filter.

0.250 is one of a long line of special-purpose materials produced by Kodak for applied photography. For details write to :

KODAK

KODAK LIMITED, DEPT. 511,
KODAK HOUSE, KINGSWAY, LONDON, W.C.2

'KODAK' is a registered trade-mark

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS
SEPTEMBER, 1950 Vol. XLII No. 251

Electronic Controller for use with High Temperature Creep Testing Equipment

By F. Gartside, A.Eng.
Research and Development Department, The United Steel Companies Limited

In the temperature controller described, modifications to the original Prosser circuit include an alteration to the layout of the Wheatstone Bridge network, provision of effective compensation for variation in the temperature of the resistance thermometer leads, and the incorporation of a visual bridge-balance indicator.

FOLLOWING the publication by Prosser*, about 11 years ago, of a circuit for an alternating-current-bridge temperature controller, a number of these controllers was constructed and found to give very satisfactory service in the control of the temperature of test specimens to within close limits for long periods of time.

Subsequently, some modifications were made to the original circuit in that the layout of the Wheatstone Bridge network was altered and an effective compensation for variation in the temperature of the resistance thermometer leads included. In addition a visual bridge balance indicator for rapidly balancing the controller was introduced into the circuit.

Experience has shown that, in addition to the high degree of accuracy of temperature control possible with this type of controller, it can also be recommended for its reliability and very low maintenance. So successful were the tests with the first few controllers that the number now in use in the author's laboratory totals more than fifty.

The use of the controller is not, however, restricted to high sensitivity creep and stress-to-rupture testing, it having been found very suitable for many other applications where it is desired to control the furnace temperature, such as in short time tensile testing, where determinations of the limit of proportionality and proof stress are made, hot fatigue tests, heat treatment, etc.

It was felt that a description of the modification made, which could not very well be undertaken without referring to the functioning of the controller as given by Prosser, would be helpful where the construction of this type of controller was contemplated. In addition, it was considered that reference to the performance of the controller when operating in conjunction with creep test equipment would also be of interest.

Method of Operation of Controller

The circuit diagram of the controller, for which the retail price of the components listed in the Appendix at the end of the paper is about £15, is given in Fig. 2 and a general view of the instrument is shown in Fig. 1. The high tension supply for operating the amplifier valves V_1 and V_2 and the electron ray indicator V_5 is obtained from a voltage doubling rectifier and smoothing circuit fed from the 110-volt winding on the mains transformer.

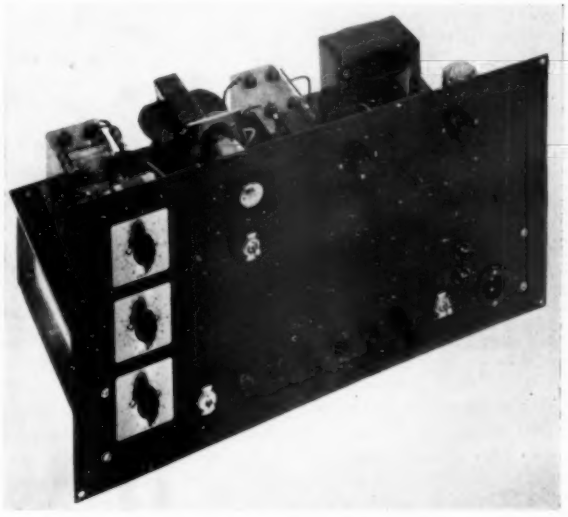


Fig. 1.—General view of controller.

In principle the control acts as a simple single-pole on-off switch actuating a main current relay. A Wheatstone Bridge circuit is employed, one arm of which consists of a non-inductive platinum resistance thermometer situated in the zone to be controlled, i.e., near to the specimen or to the winding of the furnace by which the specimen is heated. This bridge circuit is fed with alternating current at 4 volts and, by means of three potentiometers, may be balanced at any temperature within the working range which, for the particular components selected and with a thermometer resistance of 7·5 ohms at 0° C., is about 0° C. to 1,030° C.

The change in resistance of the thermometer due to temperature variations unbalances the bridge voltage. This out-of-balance voltage, after amplification by the two-stage amplifier V_1 and V_2, having a gain of about 2,000, is fed, in the correct phase, to a control valve V_3. The anode current of this valve, through reversing contacts, charges or discharges the 10 μ F condenser C_2, in the grid circuit of the relay valve V_4, which has a telephone type relay in its anode circuit.

* Alternating Current Bridge Thermostat for Creep Testing, by L. E. Prosser, B.Sc., A.K.C., *Engineering*, July 28th, 1939, p. 95.

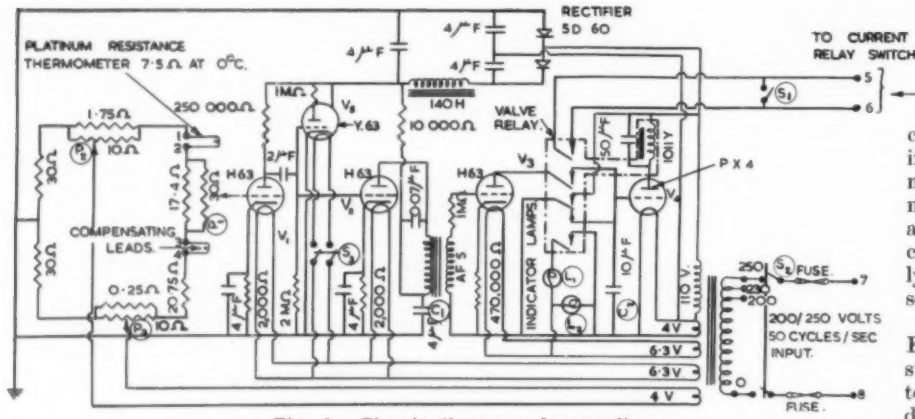


Fig. 2.—Circuit diagram of controller.

With no input from the bridge, the circuit functions in such a manner that the valve-operated relay is in the "on" and "off" positions for equal periods of time, usually about 15/20 seconds in each position, i.e., total cycle of 30 to 40 seconds. When an out-of-balance voltage from the A.C. bridge network, due to a change in temperature causing the resistance of the resistance thermometer to deviate from the value set by the potentiometers, is applied to the control valve V_3 from the amplifier circuit, there is an alteration in the rate of charge and discharge of the 10 μ F condenser and consequently the ratio of this "on" to "off" period is varied. In the circuit shown in Fig. 7 the valve-operated relay is made to actuate a single-pole current relay switch which short-circuits a series resistance in the main furnace circuit, thereby changing the effective heating of the furnace by a proportional amount to compensate for the change in temperature.

Bridge Circuit.—A circuit diagram of the Wheatstone Bridge is given in Fig. 3; the arrangement provides for simple and sensitive adjustment. Three potentiometer controls P_1, P_2 and P_3 in series with non-inductive resistances (two 30 ohm and one 20·75 ohm) are provided for balancing the bridge circuit. These are arranged to give the following approximate ranges when used with a resistance thermometer of 7·5 ohms at 0° C.

Coarse Adjustment Position Potentiometer P_1	Medium Adjustment Position Potentiometer P_2	Range D to F on Fine Adjustment Potentiometer P_3
A	G	1005° to 1034° C.
A	H	939° to 956° C.
A	I	870° to 883° C.
B	G	523° to 453° C.
B	H	464° to 482° C.
B	I	412° to 432° C.
C	G	98° to 121° C.
C	H	47° to 79° C.
C	I	1° to 33° C.

To provide the low resistance required for adjustment, and to eliminate any "step" effect, medium resistance potentiometers having a fairly large number of turns have been shunted, as shown in Fig. 3. Such an arrangement assists in the elimination of variation in contact resistance. The sliding contacts on the potentiometers are used to feed the bridge, the bridge circuit remaining complete at all times.

The fixed resistances were non-inductively wound with D.S.C. Constantan wire of 30 SWG. Provision was also made for compensation for variation in temperature of the resistance thermometer leads. The compensation is accurate if the settings of the medium and fine adjustment potentiometers, P_2 and P_3 respectively, are central, and is only slightly inaccurate for other settings.

Controls.—As shown in Fig. 1, all the necessary switches, potentiometers, terminal connections, indicator lamps and fuses have been mounted on the front panel which measures 19 in. × 10½ in. Although such an arrangement is not essential it has been found to be very convenient and assists in simplifying the layout of the wiring from the controller to the resistance thermometer in the furnace, and to the current relay switch mounted on the furnace control panel. The use of shielded wire for connecting the resistance thermometer and compensating leads between furnace and controller has been found beneficial in preventing pick-up from surrounding equipment.

The two indicator lamps L_1 and L_2 (Fig. 2) are each 8-volt, 0·1-amp. bulbs operating on a 6·3 volt supply which also supplies the filament voltage to control valve V_3. L_2 is illuminated when the control is brought into operation by the switch S_2. L_1 is illuminated when the valve relay to the control unit is in the "on" position and, provided the switch S_1 is in the "off" position, the lamp will be illuminated when the furnace is on maximum current.

In order to achieve a quick increase in temperature a single-pole switch S_1 has been connected across the leads to the current relay. With the switch in the "on" position the relevant contacts on the valve relay are short-circuited and those on the current relay switch closed. The furnace will therefore stay on maximum current irrespective of the position of the bridge potentio-

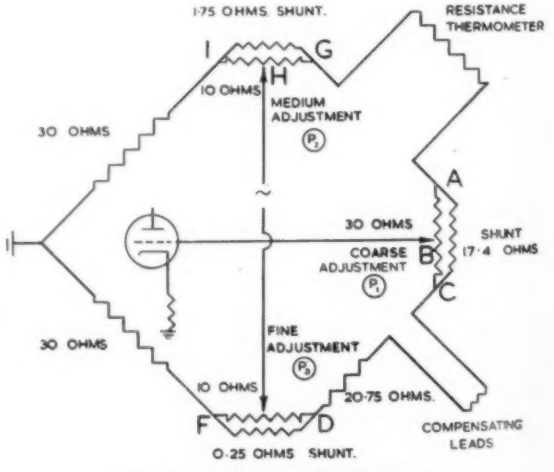
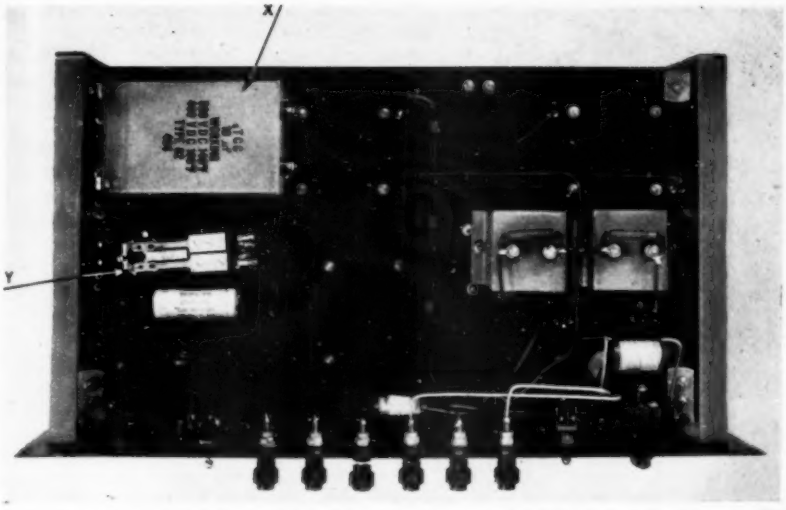
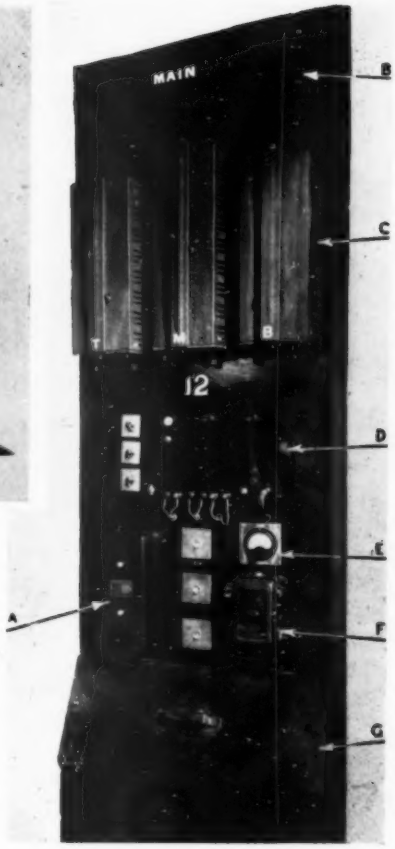


Fig. 3.—Bridge circuit diagram.



X —10 μ F RESERVOIR CONDENSER, C_2. Y —VALVE OPERATED RELAY SWITCH.

Fig. 4.—Assembly under base board.



A —CURRENT RELAY SWITCH.
B —MAIN SERIES RESISTANCE.
C —FURNACE SHUNT RESISTANCES.
D —CONTROLLER.
E —AMMETER.
F —MAIN SWITCH.
G —3·75 KVA TRANSFORMER IN BASE.

Fig. 5.—Furnace control panel.

meters P_1, P_2 and P_3 or even when the control is switched off by the switch S_2.

Having almost attained the desired temperature at which it is required to control, the switch S_1 is placed in the "off" position and left in that position at all times while the control is operating.

Visual indication of the bridge balance is provided by an electron ray indicator (Tuneray type Y63) shown at V_5 in Fig. 2, which follows the first amplifier valve V_1. V_5 is brought into operation by the switch S_3 and is intended to be used for rapidly balancing or checking the performance of the control at intervals when necessary. A small out-of-balance voltage in the bridge circuit is shown by a change in the shadow angle on the fluorescent screen. If V_5 were arranged to follow the amplifier valve V_2 instead of valve V_1, considerably smaller out-of-balance voltage on the bridge circuit would be shown for a corresponding change in the shadow angle on the indicator.

It is neither desirable nor advisable to run the indicator V_5 continuously since it decreases the sensitivity of the controller very slightly and also causes fatigue of the fluorescent screen.

Valve Operated Relay.—The 1,500-ohm telephone pattern relay, which is in the anode circuit of the relay valve V_4, as supplied, tends to stick in the half closed position. A small spring attached to two stirrups soldered on to the moving arm, and passing round the body of the relay increases the closing force and overcomes this difficulty very effectively. It is essential to keep the contacts free from dirt and with the layout of components shown in Fig. 4, a suitable position is under the base of controller.

Balancing the Control.—A condenser of suitable capacity is required across the primary of the intervalve transformer to ensure that the grid and anode voltages of the control valve V_3 are in phase, or antiphase. For this purpose the double-beam oscillograph is invaluable. The earth of the oscillograph should be connected to the cathode of the control valve and the deflector plates connected to the anode and grid of the valve, respectively. With a 15-ohm resistance in place of the resistance thermometer and the compensating leads shorted, two traces will be observed on the screen of the oscillograph when the bridge circuit is out of balance. The capacity of the phasing condenser is then adjusted to bring these traces exactly into or out of phase. The value of this condenser has been found to be of the order of 0·06 to 0·08 μ F with the particular transformer used, rather than 0·03 μ F as suggested by Prosser.

There is no guarantee that the windings on the intervalve transformer are in the same phase. If they are out of phase the control will be inherently unstable, switching on to maximum current position when the temperature of the furnace increases and on to minimum position when the temperature of the furnace falls below the control point. This can be checked by "short-circuiting" and "open-circuiting" across the resistance thermometer terminals. On "open-circuit" the valve relay should switch to the "off" position almost immediately, and on "short-circuit" it should switch to the "on" position immediately. If the reverse happens, the connections to the coupling transformer secondary (or primary), should be reversed. If for convenience, the primary connections are reversed the lead to the 4 μ F decoupling condenser C_1 should be transferred, otherwise the amplifier will not feed into the control valve.

The time period of the control may be found to be appreciably less than the stated 40 seconds total, in which case it may be increased by increasing the capacity of the 10 μ F reservoir condenser C_2 in Fig. 2 or,

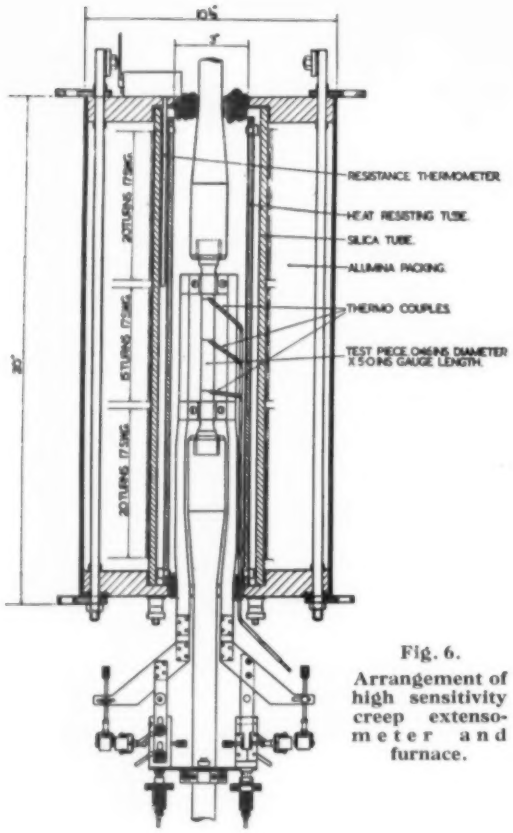


Fig. 6.
Arrangement of high sensitivity creep extensometer and furnace.

alternatively, by increasing the value of the 470,000-ohm bias resistance in the cathode of the control valve V_3. If the bias resistance is increased, care should be taken that this increase in resistance does not produce excessive feed back.

Resistance Thermometer.—The design of the resistance thermometer will depend upon the design of furnace in which it is to be used. It should be wound non-inductively, 0·25 mm. diameter commercial platinum wire being quite suitable for the purpose. If wound on double bore silica rod it is advisable to give a coating of heat resisting cement to hold the wire in position, but the coating should not be so thick as to prevent the winding from being exposed to direct radiation from the wall of the furnace. It is essential that the coating does not act as a conductor at temperature.

Current Relay Switch.—The type of current relay switch will depend to some extent on the furnace requirements, but it is essential that the load and control circuits be separated, and that the time lag in the relay is kept to a minimum in order that it does not interfere with the functioning of the controller. Standard commercial single-pole relays for alternating current, have been found to be quite suitable for this purpose. The current relay switch can be fitted into the controller or it can conveniently be mounted externally, say on a furnace control panel as shown in Fig. 5, in which case it has the advantage that where the current relay switch has to handle currents of about 10 to 12 amperes, only the leads in the control circuit of the relay switch go to the controller.

Furnace Current.—It is essential for the successful operation of this type of temperature controller that the maximum and minimum current input to the furnace be correctly chosen. The current relay switch should be arranged to increase the power input to the furnace by 50% to 100%, i.e., to vary the current in the ratio of 1 : 1·22 as a minimum to 1 : 1·42 as a maximum. The mean current should be capable of maintaining the temperature of the furnace fairly steady at the control temperature.

If these conditions of minimum to maximum currents are observed, the time periods for each half cycle should be approximately the same.

Performance of Controller

The degree of temperature control which can be attained is dependent not only upon the sensitivity of the controller but upon the design of the furnace, which in laboratory tests for creep testing purposes, etc. is generally of the electric resistance type. In Fig. 6 is shown an arrangement of a furnace together with the specimen and an extensometer used in creep testing. The disposition of the windings on the furnace tube to give uniform temperature along the specimen depends on the dimensions and materials used in construction of the furnace, and there appears to be no means other

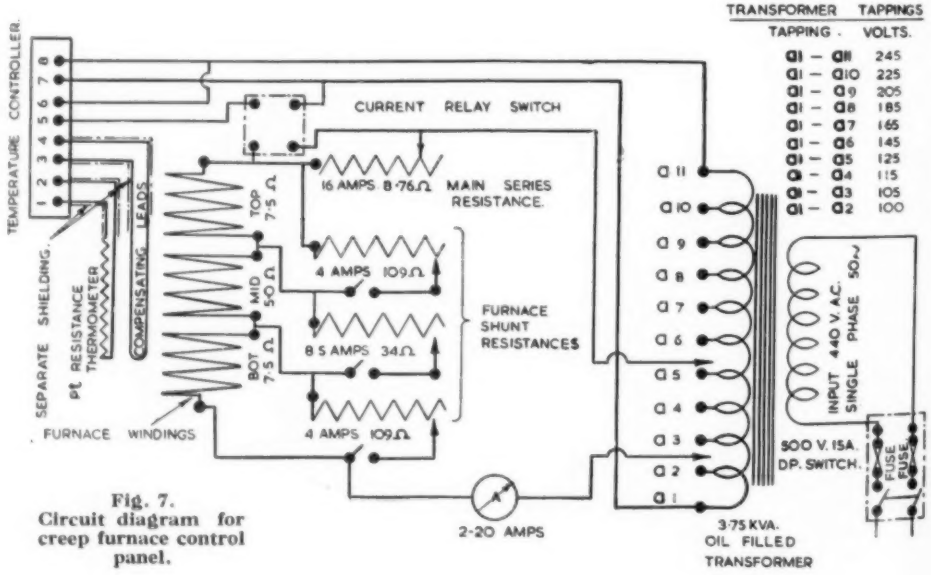


Fig. 7.
Circuit diagram for creep furnace control panel.

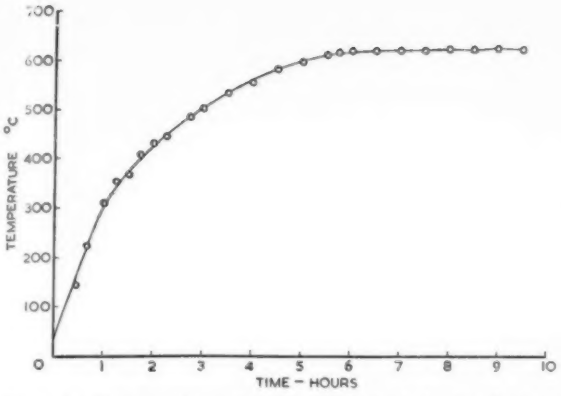


Fig. 8.—Temperature-time curve during heating-up period. Furnace voltage (max.) 145 volts.

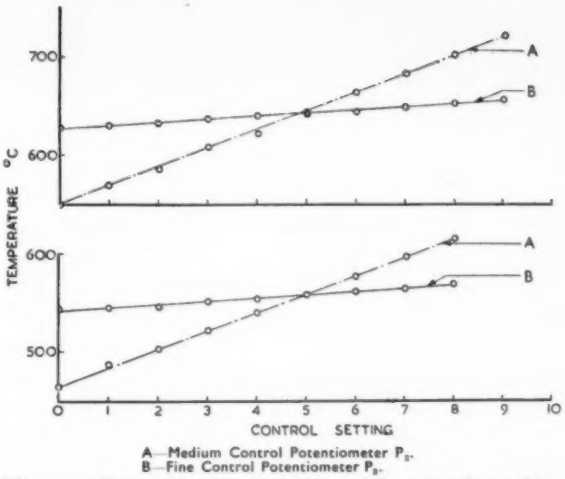


Fig. 9.—Change in temperature with control setting.

than that of experience for predetermining the correct spacing of the windings.

With a furnace of the type shown in Fig. 6 having three heating zones and a total resistance of about 20 ohms, connected in the circuit as shown in Fig. 7, the performance is such that the temperature of the test specimen can be maintained within very close limits over long time periods.

The temperature-time curve, Fig. 8, taken during the heating-up period shows a progressive increase in temperature during the first 6 hours after which there is a levelling off at a temperature of 620° C.

Ease of setting of the medium and fine potentiometers P_2 and P_3 is shown by the graphs in Fig. 9. The temperature readings were taken for different settings of each potentiometer after the furnace temperature had been allowed to become stable, first at a temperature of 645° C. and secondly, at a lower temperature of 565° C. The change in temperature for one division movement on the fine potentiometer P_3, corresponded to 3° C., and on the medium potentiometer P_2, to 19° C. for each temperature.

Behaviour of the control over long time periods is of great importance and a typical record of test specimen temperature, during a test carried out with a furnace of the type shown in Fig. 6, at a temperature of 650° C. for a period of about 1,000 hours, is shown in Fig. 10, together with the variation in room temperature which was not controlled. It will be seen that the temperature of the specimen was maintained within limits of $\pm\frac{1}{2}$° C., whilst the room temperature varied between 18·5° and 26·5° C.

As mentioned by Prosser, the sensitivity of the controller could be increased considerably but in its present form it is of ample sensitivity for the purpose of maintaining the temperature of test specimens within close limits over long periods when used in conjunction with suitably designed furnaces.

Acknowledgment

The author wishes to express his thanks to Mr. F. H. Saniter, Director of Research, Research and Development Department, The United Steel Companies Limited, for permission to publish this paper, and to his colleagues who have so willingly assisted in the construction of these temperature controllers and creep equipment.

Appendix

Components Used in Construction of Controller

Quantity	Description
1	Mains transformer. Input 200/230/250 volts. Secondary windings 110 v. 100 milliamps. 4 v. 1–2 amps. 6.3 v. 1–2 amps. 6.3 v. 1–2 amps.
1	140 Henry inductance choke.
1	Inter valve transformer.
1	"Tuneray" type 63 tuning indicator.
3	Type H.63 valves.
1	Type PX4 valve.
2	8 v. 0.1 amp. M.E.S. bulb.
1	Metal Rectifier, Voltage Doubler type, 110 volt input.
1	Telephone switchboard pattern relay wound to a resistance of 1,500 ohms and fitted with 4 sets of silver change over contacts.
	Variable Resistances (Each with escutcheon plate and knob.)
1	30 ohms, 4 watts potentiometer—P_1
2	10 ohms, 4 watt potentiometer—P_2 and P_3
	Fixed Resistances.
2	2,000 ohms, ¼ watt.
1	10,000 ohms, ¼ watt.
1	250,000 ohms, ¼ watt.
1	470,000 ohms, ¼ watt.
2	1 meg. ohm, ¼ watt.
1	2 meg. ohms, ¼ watt.

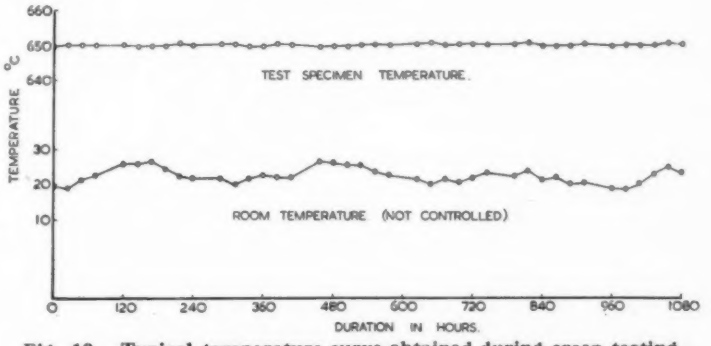


Fig. 10.—Typical temperature curve obtained during creep testing.

Quantity	Description
2	30 ohms
1	20·75 ohms
1	17·4 ohms
1	1·75 ohms
1	0·25 ohms
	Condensers
1	50 μF. (50 v. Elect.)
1	10 μF. 500 v. D.C. Working.
4	4 μF. 500 v. D.C. Working.
2	4 μF. 500 v. D.C. Test.
1	2 μF. 500 v. D.C. Working.
1	0·05 μF., Type 460
1	0·02 μF., Type 460
1	0·01 μF., Type 460

For bridge circuit, non-inductively wound with 30 S.W.G. constantan wire.

Inter-valve transformer primary condenser, capacity adjusted to obtain correct phase relationship.

Quantity	Description
	Miscellaneous
4	Octal valve bases.
1	5-pin valve base.
3	Valve top connectors.
2	Fuse holders, panel mounting.
2	1 amp., standard 1¼ in. radio fuses.
2	Type A.T.C. indicator lamps (Red M.E.S.)
2	D.P. "on-off" switches—panel mounting.
1	S.P. "on-off" switch—panel mounting.
1	3 amp. plug and socket type.
1	Voltage selector terminal strip.
3	Terminals 2 B.A.—Red.
3	Terminals 2 B.A.—Black.

Some New Indicators in Various Titrimetric Processes

By A. J. Nutten, B.Sc., A.R.I.C.

Department of Chemistry, The University, Birmingham

Recent years have seen the development of many new indicators of all types. The more important ones in the acid-base, redox, iodometric, bromometric, adsorption and miscellaneous classes are discussed in this series of articles.

DURING the past several years, numerous contributions have been made to the field of indicators. Many original titrimetric processes have been devised, and some of the older indicators have been improved.

It is proposed in this series of articles to discuss the more important of the new indicators; for purposes of convenience, they have been classified as follows:—

I.—ACID-BASE
  (a) Single: (b) Mixed: (c) Screened: (d) Universal.
II.—REDOX
III.—IODOMETRIC (and IODATE)
IV.—BROMOMETRIC
  (a) Irreversible; (b) Reversible.
V.—ADSORPTION
VI.—MISCELLANEOUS

I.

ACID-BASE INDICATORS.—(a) SINGLE TYPE

Ethyl bis (2, 4—dinitrophenyl) acetate

*p*H interval 7·5–9·1 Colourless (acid)—→Deep Blue (base)

The properties of this compound have been studied by Fehnel and Amstutz[1]. They found that it had indicator properties and was suitable for titrations usually performed with phenolphthalein. Sharp and accurate end-points were obtained in red- and orange-coloured solutions, in which the end-point with phenolphthalein was not visible.

Indicator Solution: A saturated solution of ethyl bis (2, 4—dinitrophenyl) acetate in equal parts of ethanol and acetone. (5 drops/100 ml. solution.)

The indicator may be used for titrations in anhydrous solvents, e.g., ethanol, ether and benzene, when the same reversible colour change is obtained. It is of interest to note that the indicator has been successfully used by colour blind operators who could not easily observe the end-point with phenolphthalein.

Benzoyl Auramine G

*p*H interval 5–5·6 Violet (acid)—→Pale Yellow (base)

Scanlan and Reid[2] proposed benzoyl auramine G as an acid-base indicator, ideally suited for use in Kjeldahl nitrogen determinations, in this respect being more satisfactory than either methyl red or cochineal. The shortness of the *p*H range gives a sharp colour change at the end-point.

Indicator Solution: A 0·25% solution of benzoyl auramine G in methanol. 3–7 drops per titration.

Due to slight hydrolysis of the indicator compound, the indicator should be added immediately before the titration operation is commenced. The acid form of the indicator exhibits marked dichroism. In dilute solution or in a thin layer it is blue, while in concentrated solution it is red-violet. The change to yellow is easily recognisable in daylight or under tungsten light (at a *p*H of 5·4 the indicator appears grey in daylight, and red under tungsten lighting).

Tetrabromophenol Blue

*p*H interval 3–4·6 Yellow (acid)—→Blue (base)

Bromophenol blue suffers from the disadvantage that it is dichroic. In 1929, Harden and Drake[3] described the preparation of tetrabromophenol blue, and suggested its use as an acid-base indicator. The compound is not dichroic, and has the same *p*H interval as bromophenol blue.

Indicator Solution: 100 mg. tetrabromophenol blue, 1·01 ml. 0·1 N sodium hydroxide, distilled water to 100 ml. 1–3 drops/10 ml.

Although the indicator was discovered 20 years ago and is more satisfactory than bromophenol blue, it is not yet commercially available in this country.

Emodin

*p*H interval 6·8–7·6 Yellow (acid)—→Red-Orange (base)

Emodin[4] may be used as an indicator for the titration of weak acids or weak bases. Its sensitivity is but negligibly affected by the presence of neutral salts.

Indicator Solution: A 0·1% solution of emodin in ethanol.

In the same paper, methods are given for the preparation of yellow and red emodin indicator test papers. They are as follows:—

TABLE I.

Indicator	Acid Colour	Basic Colour	*p*H Transition	Composition of Indicator	Ref.
1. Cymyl Orange	Yellow	Pink	3–4 approx.		9
2. p–Dimethylamino–p'–azobenzene–sulphonamide	Red	Yellow	3–4·5		10
3. Disodium 4, 4'–bis (p–dimethylaminophenylazo)–2, 2'–stilbene disulphonate	Orange-Red	Blue-Violet	4–5·4	0·1% aq. soln.	11
4. Disodium 4, 4'–bis (o–tolyltriazeno)–2, 2'–stilbene disulphonate	Yellow	Muddy	4–5 approx.	0·5% aq. soln.	11
5. 1–(2–hydroxy–1–naphthylazo)–2–naphthol–4–sulphonic acid	Red	Marine Blue	7–8·3		12
6. Trimethoxydihydroxy–oxo triphenylmethane (rubrophen)	(i) Cherry Red (ii) Yellow	Yellow Violet Yellow	1–3 6·5–7·2 7·6–8·2		13 14
7. 3, 4–Bis (4–hydroxy–3–nitrophenyl) hexane					
8. 5, 8–Quinolinequinone–8–hydroxyquinolyl–5–imide (indo oxine)	Red	Blue	6–8		15
9. Disodium hydroxymercuridibromofluorescein (mercurochrome)	Red	Fluorescent	4·6	1% aq. soln.	16, 17
10. p–Ethoxychrysoidine	Yellow	Red	3·5–5·5	0·2% alc. soln.	18
11. Bz–2–oxobenzanthrone	Canary Yellow	Purple	7·4	Satd/soln. in 48% alc.	19
12. Bz–2–hydroxybenzanthrone	Canary Yellow	Purple	7·4	Satd/soln. in 48% alc.	19
13. Disodium 2–(4'–nitrophenylazo)–1–naphthol–4, 8–disulphonate	Pink	Light Purple	8–10 approx.	0·1% aq. soln.	20
14. Disodium 2–(4'–nitrophenylazo)–1–naphthol–3, 8–disulphonate	Pink	Light Purple	11–13 approx.	0·1% aq. soln.	20
15. Disodium 2–(2'–methoxy–4'–nitrophenylazo)–1–naphthol–4, 8–disulphonate	Pink	Light Purple	9–11 approx.	0·1% aq. soln.	20
16. 4–Nitrocatechol	Light Straw	Lemon Yellow	5·9–6·3	1% aq. soln.	21
17. Sodium quinizarin–6–sulphonate	Yellow	Pink	7·1–9·1		22

Yellow : Place a strip of filter paper in a 0·1% ethanolic solution of emodin till thoroughly wetted. Remove and dry in the absence of ammonia vapours. The yellow colour is changed to red by 1 drop of 0·05 N alkali.

Red : Place a strip of filter paper in a 0·05 N solution of alkali. Remove and dry. Place the dried strip in a 0·1% solution of emodin in 0·05 N alkali, and dry again. The red colour is changed to yellow by 1 drop of 0·005 N hydrochloric acid.

Diphenylcarbazide

For titration of acids in dark-coloured solutions, Kröller[5] recommends the following procedure : Add 0·5 ml. ether, a few drops of a 35% aqueous formaldehyde solution and diphenylcarbazide indicator to the acid solution. Titrate the ether layer with sodium hydroxide to the first colour change.

Indicator Solution : A saturated solution of diphenylcarbazide in methanol. 4–5 drops per titration.

Lead Acetate, Dithizone

Transition : *p*H 4 Green (acid)⟶Red (base)

A novel procedure for indicating the end-point in acid-base reactions has been discussed by Hirsch.[6] To the aqueous solution to be titrated, he added a small amount of lead acetate and a solution of dithizone in an organic solvent. If the *p*H of the solution was less than 4, most of the lead remained in solution and the colour of the organic solvent layer was green, due to the dissolved dithizone. If the *p*H was greater than 4, the organic solvent extracted some of the lead, and became red in colour due to the formation of lead dithizonate.

Indicator Solutions :

(i) A dilute solution of lead acetate in water.

(ii) 50 mg. dithizone dissolved in 1 litre of carbon tetrachloride, chloroform, benzene, amyl alcohol or any other suitable solvent.

A few drops (i) and a few ml. (ii) per titration.

Titration is carried out with 0·1 N sodium hydroxide, with shaking after each addition. Weak acids cannot be titrated in this way. It may be possible, according to Hirsch, to use the indicator in the titration of coloured aqueous solutions if the colour is not extracted by the organic solvent. Kolthoff[7], however, states that the indicator is of little practical use for this purpose, because traces of various metals in the aqueous solution would interfere.

Trivalent Thallium Salts

Colourless (acid) ⟶ Yellow (base)

Trivalent thallium salts are claimed to be satisfactory as internal indicators in acidimetry. Zolotukhin[8] has described their use in titrations involving sulphuric, nitric, and hydrochloric acids, in the presence of titanous chloride, and has obtained results in close agreement with those found using methyl orange as indicator.

The one advantage which these indicators appear to possess over most organic indicators is that they can be applied in the presence of chloride, bromide, hypochlorite, hypobromite and nitrite. The method of back-titration has to be used for the determination of bases and, where possible, other proved indicators should be used.

Other Acid-base Indicators

Several other acid-base indicators have been proposed during recent years. The most promising of these have been tabulated (see Table I) and notes pertaining to their use appended below.

Notes on Table I

Indicator. *Comment.*

1. Claimed to be superior to methyl orange, giving a more distinct colour change.
2. Passes from the red to the yellow form without first passing through an orange colour.
3. Suitable for yellow-coloured solutions where the bluish-violet end-point colour is reflected back as a deep green. 2 drops/100 ml.
4. Can be used in the titration of very dilute sodium carbonate solutions. Titration best carried out in a white porcelain dish to enhance the slightest variation in shade. 5 drops/100 ml.
6. Suitable for titration of very weak bases or of strong acids and bases ; or even of weak and strong bases in presence of each other.

7. In colourless solutions is suitable for *p*H measurements, its yellow colour increasing progressively through *p*H 7·6–8 to *p*H 8·2, where it remains unchanged.
8. Since the colour change is sharp and in the neutral range, it can be used for the titration of 0·01 N mineral acid with 0·01 N alkali hydroxide solutions which are not easy to titrate with most indicators.
9. To be used preferably in the titration of basic solutions. Its fluorescence in alkaline solution almost disappears at *p*H 4·6–4·5. At the end-point, a red precipitate is formed which colours the solution a light pink. 1 drop per titration.
10. 1 drop/50 ml. solution.

(b) MIXED TYPE

Several advantages may be derived from the use of mixed indicator solutions. In acid-base titrations, for example, the end-point is more distinct when 2 indicators with complementary colours, which change from neutral to the colour on the acid or alkaline side, are combined. Again, it is often possible, by varying the relative amounts of the indicators, to obtain a sharper end-point colour than that resulting from the use of a single indicator.

Bromocresol Green—Methyl Red

Bluish-Green (base) —→ Pink (acid)

A mixture of bromocresol green and methyl red has been used as an indicator in Kjeldahl nitrogen determinations by Ma and Zuazaga[23]. The indicator gives a bluish-purple colour in 2% boric acid solution, the colour changing to a bluish-green in the presence of a trace of ammonia, and to pink with a trace of mineral acid. The intensity of the colour varies but little with the amount of indicator added, and the transition at the end-point is very sharp and distinct.

Indicator Solution :

(i) 0·1% solution of bromocresol green in 95% ethanol.

(ii) 0·1% solution of methyl red in 95% ethanol.

Mix 10 ml. (i) with 2 ml. (ii). 4 drops/5 ml. 2% boric acid.

Procedure.—Titrate the boric acid solution of ammonia with 0·01 N hydrochloric acid from a bluish-green till the blue colour disappears, or to a faint pink, subtracting in the latter case 0·02 ml. from the acid reading.

The indicator possesses several advantages over other " Kjeldahl " indicators :—

(1) Acid or alkaline contamination in the titration flask is immediately discovered on introduction of boric acid and indicator.

(2) If the boric acid does not change to bluish-green with the first drop of distillate, either the sample is nitrogen-free, or the digestion mixture has not been sufficiently neutralised with sodium hydroxide.

Bromocresol Green—Methyl Yellow

Yellow (acid) —→ Blue (base)

Hoppner[24] used a mixture of bromocresol green and methyl yellow as a substitute for methyl orange.

Indicator Solution :

(i) 0·2% solutior of bromocresol green in ethanol.

(ii) 0·2% solution of methyl yellow in ethanol.

Mix 4 parts (i) with 1 part (ii). 8 drops/100 ml, solution.

The colour of the mixed indicator changes sharply from blue in alkaline solution to yellow in acid, the recognition of the end-point being easy because of the sudden change from a dark to a light shade. The indicator is especially suited for use in the titration of coloured solutions.

Miscellaneous Mixed Indicators

Hähnel[25] has studied many combined indicators for acid-base titrations, the most suitable being listed below :—

(i) 5 parts dimethyl yellow, 3 parts methylene blue.
(ii) 1 part methyl red, 1 part bromocresol green.
(iii) 5 parts neutral red, 2 parts methylene blue.
(iv) 5 parts neutral red, 2 parts tetrabromophenol blue.
(v) 3 parts phenol red, 2 parts bromothymol benzene.
(vi) 3 parts cresol red, 1 part thymol benzene.

The components of (v) and (vi) may not be readily obtainable, in which case the mixed indicators :

(vii) 3 parts phenol red, 4 parts bromothymol blue.
(viii) 4 parts thymol blue, 1 part cresol red, respectively, may be used.

For the titration of strong acids with strong or weak bases, indicator (i) should be used, and for alkali with acids, indicator (ii) should be used.

For general titrations in aqueous solutions the most suitable indicators are (iii) and (iv), and for ethanolic alkali (up to 70% ethanol) with acids, indicator (iv).

For the determination of acidity and ester control of ethanol (70%) the best results are obtained with indicator (v), and for the titration of ethanolic alkali with biphthalate, indicator (vi) is satisfactory.

(c) SCREENED TYPE

The addition of a dyestuff to an acid-base indicator may improve the colour change at the end-point. This improvement is often due to the fact that the modifying dyestuff filters out a portion of those wavelengths not appreciably absorbed by either form of the indicator. Although the end-point colour may be completely different from that of the unscreened indicator, the *p*H interval over which the colour transition takes place remains unaltered.

Methyl Yellow—Methylene Blue

*p*H interval 2·9–4 Yellow-Green (base)—→Pink (acid)

It is well known that peculiar difficulties attend the titration of sodium carbonate with hydrochloric acid, due mainly to the buffer effect of the solution on either side of the end-point—this buffer action having a marked effect on the response of the indicator to additions of acid. Carmody[26] has successfully used a mixture of methyl yellow and methylene blue as indicator for the direct titration of sodium carbonate solutions.

Indicator Solution : 0·8g. methyl yellow and 0·04g. methylene blue dissolved in 1 litre of ethanol.

0·1 ml./100 ml. volume at end-point.

The indicator is coloured yellow-green in alkaline solution ; as the end-point is approached this changes to a straw colour, then to a sunburned straw, and finally, at the end-point, to a pink-straw. The end-point is taken as the first trace of pink. It is preferable to defer addition of the indicator till the titration is more than half completed, as the methyl yellow fades rapidly in alkaline solution.

Carmody gives absorption curves to indicate the superiority of methyl yellow over methyl orange and the suitability of using methylene blue as a colour-modifying substance.

Sodium Salt of Methyl Red—Alphazurine

*p*H interval 4·8–4·6 Green-Grey (base)——→Purple-Grey (acid)

The above screened indicator was introduced by Fleisher[27] for the determination of alkalinity in boiler feed water. With its use, it is possible to titrate 0·003 M sodium carbonate with 0·033 N hydrochloric acid.

Indicator Solution : 0·45 g. sodium salt of methyl red and 0·55 g. alphazurine dissolved in 1 litre of distilled water.

The colour changes are claimed to be sharp and distinct, and to show sufficient contrast to make the changes easily distinguishable. A grey intermediate colour gives ample warning of the approaching purple-grey which is used as the end-point of the titration.

Tropolein OOO No. 1—Malachite Green

Krishnamurty[28] tested several mixed indicators for the titration of CO_3^{-2} to HCO_3^{-1} with hydrochloric acid. He found that a screened indicator consisting of a 1 : 1 mixture of Tropolein OOO No. 1 and Malachite Green gave the best end-point.

(d) UNIVERSAL TYPE

(1) Dubsky and Langer [29] have described 2 universal indicators for estimating *p*H in the limits 1—7 and 7—14, respectively. Each indicator possesses a change in colour in the succession of the spectrum from red to blue, and a distinct shade of colour from *p*H 1 to 2, etc.

Indicator Solutions :

(i) 0·35g. thymolsulphophthalein,
0·2g. tropaeolin 00,
0·1g. tetrabromophenolsulphophthalein,
0·3g. bromocresol green,
0·4g. bromocresol purple,
dissolved in 1 litre of 50% ethanol.

(ii) 0·35g. neutral red,
0·15g. thymolsulphophthalein,
0·25g. thymolphthalein,
0·1g. nitramine,
0·6g. m—nitrophenol,
dissolved in 1 litre of 50% ethanol.

(2) A universal indicator for determining *p*H in the range 1·2–12·7 has been proposed by Cuta and Kamen[30]. They prepared a mixture of indicators, containing in 1 litre of ethanol :—

0·125g. s—trinitrobenzene,
0·0355g. phenolphthalein,
0·03g. cresolphthalein,
0·1g. dibromothymolsulphonephthalein,
0·022g. methyl red,
0·0085g. methyl orange,
0·05g. pentamethoxyl red.

The colour changes take place in the *p*H ranges 14–12, 10–8·3, 9·8–8·2, 7·6–6, 6·3–4·2, 4·4–3·1 and 3·2–1·2, respectively. In 96% ethanolic and aqueous solution, the pentamethoxyl red and the bromothymol blue interact and precipitate as a smudge.

The universal indicator is stable in anhydrous ethanol or in methanol. Since most of the individual indicators are more soluble in methanol than in ethanol, the former solvent is preferable.

(3) Van der Burg[31] has described tests with a universal indicator, which gives the following distinct colour changes at whole *p*H numbers between 4 and 10 : red, orange, yellow-green, blue, indigo, violet.

Intermediate colour changes are recognisable with an accuracy of 0·5 *p*H.

Indicator Solution :

5 mg. thymol blue,
25 mg. methyl red,
60 mg. bromothymol blue.
60 mg. phenolphthalein,

dissolved in 75% ethanol to make 100 ml. solution. This solution is neutralised with 0·01 N sodium hydroxide solution to produce a green colour, but for high and low *p*H it can be used directly.

REFERENCES.

1 Fehnel, E. A., and Amstutz, E. D., *Ind. Eng. Chem.* (*Anal. Ed.*), **16**, 53 (1944).
2 Scanlan, J. T., and Reid, J. D., *Ind. Eng. Chem.* (*Anal. Ed.*), **7**, 125 (1935).
3 Harden, W. C., and Drake, N. L., *J. Am. Chem. Soc.*, **51**, 562 (1929).
4 Umanskii, Z. M., *Zavodskaya Lab.*, **11**, 404 (1945).
5 Kröller, E., *Deut. Leb. Runcbchar*, **44**, 31 (1948).
6 Hirsch, W., *Analyst*, **73**, 160 (1948).
7 Kolthoff, I. M., *Anal. Chem.*, **21**, 101 (1949).
8 Zolotukhin, V. K., *Zavodskaya Lab.*, **9**, 133 (1940).
9 Wheeler, A. S., and Waterman, J. H., *J. Elisha Mitchell Sci. Soc.*, **49**, 36 (1933).
10 Lente, K. van, and Pope, G., *Trans. Illinois State Acad. Sci.*, **39**, 77 (1948).
11 Taras, M., *Anal. Chem.*, **19**, 339 (1947).
12 Singu, H., and Masuo, H., *J. Chem. Soc. Japan*, **60**, 595 (1939).
13 Szebellédy, L., and Ajtai, M., *Magyar Gyogyszeresztud. Tarsasag. Ertesitoje* **13**, 822 (1937).
14 Spitzer, L., *Ann. chim. applicata*, **32**, 180 (1942).
15 Berg, R., and Becker, E., *Z. anal. Chem.*, **119**, 81 (1940).
16 Airan, J. W., *Nature*, **160**, 88 (1947).
17 Domange, L., *Ann. pharm. franc.*, **2**, 55 (1944).
18 Schulek, E., and Rozsa, P., *Z. anal. Chem.*, **115**, 185 (1939).
19 Fish, F. H., and Wrenn, W. H., *Virginia J. Sci.*, **3**, 12 (1942).
20 Ferber, K. H., *Ind. Eng. Chem.* (*Anal. Ed.*), **18**, 631 (1946).
21 Cooper, S. R., and Tulane, V. J., *Ind. Eng. Chem.* (*Anal. Ed.*), **8**, 210 (1936).
22 Green, J. H., *Ind. Eng. Chem.* (*Anal. Ed.*), **14**, 249 (1942).
23 Ma, T. S., and Zuazaga, G., *Ind. Eng. Chem.* (*Anal. Ed.*), **14**, 280 (1942).
24 Hoppner, K., *Deut. Zuckerind*, **61**, 361 (1936).
25 Hähnel, S., *Svensk Kem. Tids.*, **47**, 4 (1935).
26 Carmody, W. R., *Ind. Eng. Chem.* (*Anal. Ed.*), **17**, 141 (1945).
27 Fleisher, H., *Ind. Eng. Chem.* (*Anal. Ed.*), **15**, 742 (1943).
28 Krishnamurty, K. V. S., *J. Indian Chem. Soc.*, **25**, 492 (1948).
29 Dubsky, J. V., and Langer, A., *Chem. Obzor.*, **11**, 29 (1936).
30 Cuta, F., and Kamen, K., *Chem. Listy*, **30**, 22, 192 (1936). *Collection. Czechoslov. Chem. Communs.*, **8**, 395 (1936).
31 Burg, J. H. N. van der, *Chem. Weekblad.*, **36**, 101 (1939).

Colorimetric Determination of Phosphorus in Phosphor Copper

By George Norwitz* and Irwin Norwitz*

PHOSPHOR copper is an alloy containing about 10% phosphorus and 90% copper, which is used in deoxidising bronzes. The methods which have been proposed for the analysis of this type of material are tedious gravimetric procedures[1].

It occurred to the authors that a colorimetric method might be used advantageously in determining phosphorus in phosphor copper. At first, attempts were made to use the phosphovanadomolybdate colorimetric method[2], but the results obtained were not sufficiently accurate and precise. The authors then turned to the molybdenum blue method for phosphorus, described by Hague and Bright[3] and Katz and Proctor[4]. It was found that this method would give excellent results for phosphorus in phosphor copper after the sample was dissolved in nitric acid and fumed with perchloric acid. Nitric acid, rather than a mixture of nitric and hydro-

* Laboratory of George Norwitz, 3353 Ridge Avenue, Philadelphia 32, Pa.

1 Lunge, G., and Keane, C. A. "Technical Methods of Chemical Analysis." Vol. II, p. 214, London, England, Gurney and Jackson, 1928.

2 Snell, F. D., and Snell, C. T. "Colorimetric Methods of Analysis." p. 672, New York. D. Van Nostrand Co., 1949.

3 Hague, J. L., and Bright, H. A. *J. Research Nat'l Bur. of Standards*, 26, 405 (1941).

4 Katz, H. L., and Proctor, K. L. *Anal. Chem.*, 19, 612 (1947).

TABLE I.

RESULTS FOR PHOSPHORUS IN A TYPICAL PHOSPHOR COPPER

Phosphorus Present*	Phosphorus Found
%	%
9·92	9·98
—	9·95
—	9·97
—	9·93

* As determined by gravimetric phosphate method[1].

chloric acids, must be used in dissolving the sample, since there is danger of losing phosphorus as the chloride, if hydrochloric acid is present in dissolving phosphor copper[1]. Because of the small aliquot taken copper does not interfere. The small amount of tin found in phosphor copper (less than 0·1%) causes no difficulty. Up to 0·4% of tin can be kept in solution in a perchloric acid medium.

Special Reagents

Hydrazine Sulphate Solution (0·15%).—Dissolve 1·5 gram of hydrazine sulphate in 1 litre of water.

Ammonium Molybdate Solution (2% *in* 11 *N* H_2SO_4).—Add 300 ml. of sulphuric acid to 500 ml. of water. Cool and add 20 grams of $(NH_4)_6\ MO_7O_{24}.\ 4\ H_2O$. Dilute to 1 litre.

Ammonium Molybdate—Hydrazine Sulphate Reagent.—Add 125 ml. of ammonium molybdate solution and 50 ml. of hydrazine sulphate solution (0·15%) to 275 ml. of water. Dilute to 500 ml. with water. This solution is unstable and should be freshly prepared just before using.

Sodium Sulphite Solution.—Dissolve 100 grams of $Na_2\ SO_3$ in water and dilute to 1 litre.

Standard Phosphorus Solution.—Dissolve 0·4263 gram of $(NH_4)_2\ HPO_4$ (obtained from a fresh unopened bottle) in water, and dilute to 1 litre in a volumetric flask. Each ml. of this solution contains 0·1 mg. of phosphorus.

Procedure

Add 20 ml. of nitric acid (1 to 1) to a 0·5 gram sample, contained in a covered 250-ml. beaker. Warm on the steam bath until the sample is in solution. Add 10 ml. of perchloric acid (70%), evaporate to fumes of perchloric acid, and fume for a few minutes to drive off all the nitric acid. Cool, transfer to a 250-ml. volumetric flask and dilute to the mark. Pipette a 15-ml. aliquot into a second 250-ml. volumetric flask and again dilute to the mark. Pipette out a 15-ml. aliquot into a 250-ml. Erlenmeyer flask. Add 3·0 ml. of perchloric acid (70%). and add 15 ml. of sodium sulphite solution (10%). Heat the solution to boiling and boil for 20 to 30 seconds. Remove from the hot plate and immediately add 50 ml. of ammonium molybdate-hydrazine sulphate reagent. Heat on the steam bath for 20 minutes. Cool rapidly to room temperature and dilute to 100 ml. in a volumetric flask. Read the transmittency at 690 millimicrons in a photo-electric colorimeter (1 cm. cell) that has been set to zero with a reagent blank. Convert the readings to per cent. phosphorus by consulting a curve prepared from standard phosphorus solution.

Results

The results obtained for phosphorus in a representative phosphor copper are shown in Table I. The method shows good agreement with the ammoniacal ammonium phosphate method (double precipitation)[1].

Correspondence

Reproducibility of Analytical Results

The Editor, METALLURGIA.

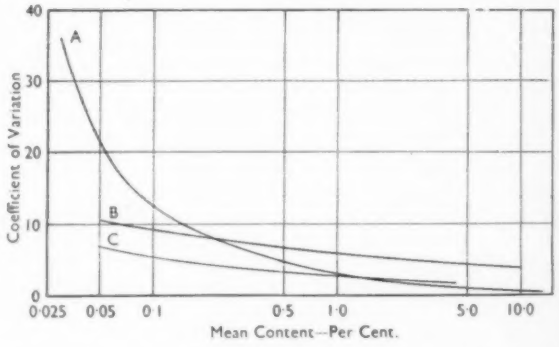
Sir,

Two most interesting articles have appeared in your journal, one by Scheuer and Smith (1949, **41**, 44–46) and the other by van Someren (1950, **42**, 52), showing a form of correlation between the reproducibility of routine estimations and the percentage concentration of the element being determined.

In our laboratory, for each batch of foundry control determinations, for any particular element, a standard sample is processed and the result recorded in the normal manner. The routine figure on the standard sample is compared with the accepted standard figure and the comparison used to detect significant errors at an early stage. Figures derived in this manner covering about a year in the case of spectrographic and two years in the case of all other methods of analysis and including those of both skilled and unskilled assistants, were considered, and graphs constructed showing mean content against " coefficient of variation " (standard deviation in percent. of mean content).

The accompanying graphs are constructed mainly from data concerning aluminium alloys, but where magnesium alloys have been analysed by any of the methods, these results have fitted quite satisfactorily into the general scheme of things and are incorporated in the final graphs given. Curve A refers to data derived from classical wet methods of analysis, curve B refers to spectrographic analysis and curve C to photometric and polarographic analysis.

We confirm the approximate form of the spectrographic curve as given by Scheuer and Smith ; however,



on working out the values to construct the graphs, it was noticed that the rate of decrease in precision for contents below about 1 per cent. was about the same for photometric and polarographic methods of analysis as for spectrographic methods, these being considerably less than for the classical wet methods (gravimetric and volumetric). For this reason it was thought useful to plot A and C separately. Hence it is seen that, in our laboratory, the precision obtained using photometric and polarographic procedures for element concentrations of below about 0·5% is measurably greater than that obtained by classical wet methods of analysis.

It would be interesting to learn if other laboratories have found such a trend with methods using instrumentation.

Yours faithfully,

S. E. HERMON.
E. C. MILLS.

Research Laboratories,
High Duty Alloys, Ltd.,
Slough, Bucks.
August 29th, 1950.